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Survey and Study on Sand and Dirt

by
Edward Kuletz
and
Howard C. Schafer
Propulsion Development Department

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ABSTRACT

This report describes the techniques of sampling and analyses of worldwide soil samples. The results demonstrate that there are considerable differences between the actual sand and dirt environment and that specified in the Military specification. Field reports show that many military and military-support items did not perform as expected even though they had fulfilled the specification requirements. Therefore, the Military Specification on Sand and Dirt should be revised to conform to the actual environmental test conditions.

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Naval Weapons Center

AN ACTIVITY OF THE NAVAL MATERIAL COMMAND

W. J. Moran, RADM, USN Commander

H. G. Wilson Technical Director

FOREWORD

This report covers progress on work conducted from 1966 to the present to determine worldwide sand and dirt environments of ordnance and military equipment. This work was supported by the Naval Air Systems Command under AirTask F19-332-301 in conjunction with other ongoing projects as a minimum cost function.

This report has been reviewed for technical accuracy by Warren W. Oshel.

Released by
CRILL MAPLES, *Head*
Quality Assurance Division
15 July 1971

Under authority of
G. W. LEONARD, *Head*
Propulsion Development Department

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<p>This report describes the techniques of sampling and analyses of worldwide soil samples. The results demonstrate that there are considerable differences between the actual sand and dirt environment and that specified in the Military specification. Field reports show that many military and military-support items did not perform as expected even though they had fulfilled the specification requirements. Therefore, the Military Specification on Sand and Dirt should be revised to conform to the actual environmental test conditions.</p>		

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Environmental Criteria Worldwide Soil Samples Analytical Results Military Specifications Sand and Dust						

INTRODUCTION

Environmental sand and dirt criteria are major factors that must be considered in the effective design of all types of ordnance material. Many military, or military-support items, have failed to withstand the harsh, realistic dynamic sand and dirt environmental demands encountered during operational usage even though they have previously satisfied the requirements as set forth in the Military Specifications (MIL-STD-810B, etc.). Therefore, it is important that a thorough investigation be made of the actual sand and dirt environment to which the ordnance materials and the transporters are subjected. The accomplishment of this type of study can then be used to make more realistic the criteria set forth in the Military Specifications. This report is concerned only with the mechanical effects of the dirt environment. The chemical and corrosive aspects need to be addressed in other work.

In support for the above goal, 264 samples of dirt have been collected from widely scattered locations around the world. In many cases, the same area was represented by a number of samples. Analytical data have been obtained on 43 dirt samples. It was possible to arrive at some generalized conclusions and to observe a number of anomalies. Visual observation of the remainder of the worldwide dirt samples (Appendix A) tend to support the conclusions herein reached, based on the analyses of the 43 samples.

PHYSICAL RELATIONSHIPS

Before a discussion of the analysis associated with sand and dirt, there are some extenuating circumstances that are not well known and should be explained. Also, the sand and dirt phenomena should not be associated only with the typical desert areas but should also include temperate and tropical areas. Practically all areas have dry periods, whether extended or localized short-duration type, when the sand and dirt "play havoc" with ordnance items and various types of ground and air transporters (Ref. 1, 2, 3). For discussion purposes, and for associating the environmental forces with the specifications, consideration will be given to the situation (dry or desert-like climate) during which the most adverse reactions occur.

DESERT PAVEMENT

In the semiarid regions of the world the soil seems to exhibit a greater tendency to stick together or agglomerate more at the surface layer than in the subsurface layers. This agglomeration of the surface layer, referred to as the "desert pavement", is in evidence extensively

in all desert or dry areas worldwide. It has been observed to exist not only at China Lake in the Mojave Desert, but also in the Philippine Islands and throughout Southeast Asia during the dry season.

The desert pavement generally consists of a surface layer 1/4 to 2 inches thick. The mechanical strength of this layer varies with the consistency of the soil, but in general it is not able to withstand the weight of modern military vehicles.

For clarification, the analogy of a frozen lake can be used. The layer of ice on the surface is the equivalent of the desert pavement. The ice usually cannot support too much weight before it is fragmented. The ice suppresses waves and holds down the water spray, which is equivalent to the desert pavement suppressing the blowing of sand.

The profuseness of a sandstorm for a given wind velocity varies drastically with the amount of disturbance to which the desert pavement has been subjected. If it is still intact, the wind must exhibit extremely high relative velocities to raise a dust haze. If the desert pavement has been totally destroyed, a zephyr is sufficient to cause the formation of a dust haze.

This phenomenon can be observed at any new housing subdivision throughout the southwestern United States. From the time of the first ground breaking, through the time when lawns or concrete take the place of the desert pavement, the dirt will permeate into everything when the wind is blowing. At this same time, the indigenous desert may be crystal clear. The contrast is striking when it is observed from one of the foothills usually surrounding the desert valleys.

MILITARY APPLICATION

The desert pavement phenomenon is merely of passing interest until it is applied to ordnance and the military in general.

When an area is chosen as a logistic supply point, the first thing that must be done is to move men and material into it by the use of vehicles, such as the workhorse 6 x 6 trucks. These vehicles proceed to shuttle supplies and equipment into the area at a tremendous rate. If one observes the tracks that serve as roads, he would notice that instead of a granular surface with spasmotic vegetation interspersed throughout, the roadway consists of a fine, sometimes fluidized, dry powder. In some locations, the dry powder will react as if it were a fluidized bed, in that it exhibits a reaction resembling a water splash when it is stepped into. When a vehicle is driven through the dry powder, even at a speed of 15 miles per hour, a dust cloud is forced into the atmosphere to a height of about 25 feet. The constant movement over the roads and in the storage area soon divest the general area of vegetation and the desert pavement.

EXAMPLE AT DA NANG

The forward Air Force--Marine Air Base at Da Nang in Northern South Vietnam will be used as an example because this area is of more interest at the present time than the pure desert. However, the facts as displayed are equally applicable to desert regions of the world.

Figures 1 through 6 are indicative of the actual situation at a forward logistical supply point. The first sequence (Fig. 1 through 3) is representative of the usual situation. The locale was the rear area of the Marine Air Wing's ordnance storage area. The road was a well-maintained, little used, secondary path. It was requested that the driver proceed at a constant 15 mph. The time of the year was 24 June 1966, which was the dry season at Da Nang.

Notice the progressive visual blockage of the surrounding terrain as the truck nears the camera station. In Fig. 1, the dust plume appears to be approximately twice the height of the trucks and three times the width. In Fig. 2 and 3, the length of the dust plume is more evident. It is obvious that any ordnance carried in the bed of the truck would be immediately enveloped by the fine silt-like dirt.

Of side interest, notice the visual blockage of the terrain in the center right-hand side of Fig. 1. The reason for this is shown in sequence 2 (Fig. 4 through 6).

This sequence is indicative of the conditions on a well traveled portion of dirt road. The location is the entrance of the Marine Air Wing ordnance storage dump at Da Nang. The trucks are required to slow down, from the 15 mph maximum speed, while clearing the "magazine" area. Even though the trucks are geared down for a slow exit, the dirt plume is impressive. This area is represented by the "blocked out" background in Fig. 1. The extent of the dirt plume is evident in Fig. 5 and 6. Notice that in Fig. 6, the plume hides the truck completely.

A closer look at the ground in the second sequence reveals a measure of the depth of this fine dirt layer. Notice that the full imprint of the 6 x 6 trucks nondirectionally treaded tire is evident in the "fluidized" dirt bed (Fig. 4). The depth of dirt was estimated to be about 3 inches. The depth of tread of the cleats is about 1 inch, depending on the age and previous use of the tire. In the rainy season the area is a quagmire. Anything handy is thrown into the roadbed to stabilize the soil. A compass will "go wild" at this location because of all the sunken metal used to stabilize the magazine entrance area.

Everything at Da Nang was covered with red dirt. The normally white paint of the Navy LAU-10 Zuni launcher was a nominal brown-pink, due to the dirt layer. An attempt was made to wipe this layer off, and it was found that the finer particles had permeated the surface of the paint to become part of the coating. (A single abandoned LAU-10 Zuni launcher is visible in Fig. 4, in the left-hand center.)



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FIG. 1. Marine 6 x 6 Truck at 15 mph Velocity on
Typical Well Maintained Road.



LHL 133764

FIG. 2. Marine 6 x 6 Truck at 15 mph Velocity on
Typical Well Maintained Road.



LHL 133766

FIG. 3. Marine 6 x 6 Truck at 15 mph Velocity on Typical Well Maintained Road.



LHL 133765

FIG. 4. Marine 6 x 6 Truck at Less Than 15 mph Velocity on Well Traveled Section.



LHL 133758

FIG. 5. Marine 6 x 6 Truck at Less Than 15 mph
Velocity on Well Traveled Section.



LHL 133762

FIG. 6. Marine 6 x 6 Truck at Less Than 15 mph
Velocity on Well Traveled Section.

The comment could be projected, at this point, that the above observations pertain only to transportation; however, this is not the case. In fact the relevance of the observation equally covers aircraft, helicopters, trucks, storage, and usage of any item of combat utility.

Figure 7 is a typical dump storage revetment. In this situation, however, the revetment dates back to the occupation of French Indochina by the French. Therefore, the vegetation is profuse on the walls of the revetment and this growth will tend to hold the dirt from blowing away. Notice the depth of the nondirectional tire tracks in the foreground. When ordnance is unloaded or retrieved here, the dust clouds are severe.

Figure 8 shows a typical ordnance buildup area. The Marines are assembling a 500 lb Mk 82 bomb and Snakeye fin assembly. Again, notice the condition of the dirt in the foreground. The troops gave a cry of anger when the truck was driven into the revetment area for this picture.

In Fig. 8, one can observe that the assembly operation suffers also. The Snakeye fin assembly, being worked on by the Marine in the white shorts, is in the process of falling to the ground. The Marine was attempting to break the corrosion bond and the dirt-cake that impeded the easy removal of the assembly from the container frame. This type of problem could be alleviated to a large extent if, during the design phase, a more careful analysis was made of the eventual operational environmental parameters.

ACQUISITION OF SOIL SAMPLES FROM AREAS OF INTEREST

To ensure that equipment and ordnance items give the maximum service life and effectiveness possible, they must be qualified under stress conditions similar to those encountered in actual use. This requires the gathering and analysis of soil samples from all possible areas of the world. The sampling priorities were established arbitrarily. The Southeast Asia--Western Pacific area had first priority because of US involvement in Vietnam. Desert areas were given second priority because of the known problems commensurate with these regions. The other samples were taken at random by personnel who were personally interested in the study and who wished to contribute to the program while they were on official and nonofficial travel. In general, any region exhibiting red soil was of interest, since this may indicate the presence of iron oxide.

Over 260 worldwide soil samples have been collected and are stored at NWC. The collection came from areas within the Pacific Ocean's "ring of fire", Far East, Europe, and the Middle East.



FIG. 7. Typical Dump Storage Revetment.



FIG. 8. Typical Dump Storage Assembly Area.

The locations of the soil sampling sites are shown in Fig. 9. The sources of the analyzed samples are shown in Table 1 and the sources of the additional samples are listed in Appendix A.

SOIL SAMPLING TECHNIQUE

For this study it was necessary to collect as many representative samples of native soil as possible. The methods used in sample taking and handling were not sophisticated, but were simplified to permit the acquisition of many samples by a variety of personnel who were not trained specifically for the task. This was feasible since the samples were to be obtained for physical and chemical analyses and not for microbiological analyses and research whereby the sampling utensils and containers must be sterilized before each use. Primarily, the samples were to be collected from areas of current military involvement. However, samples were also obtained from other areas so that a better and more complete understanding of the problems would be possible.

The majority of the samples were obtained by personnel who were on official travel; this was accomplished at virtually no added expense or inconvenience. In general, the participants were instructed to scoop some soil into a container and then send it to China Lake. The exact place or sample depth was of small consequence, since wherever the sample was obtained, it would be representative of the general area. The general area should include, if possible, that portion of the country where sand and dust conditions were most severe. If an anomalous situation was uncovered, it would eventually, with enough general samples, become properly classified.

The sampling container was chosen which would withstand handling by postal personnel and yet safely contain potentially harmful microscopic life. It consisted of a quart-size paint can outside shell and a polyethylene bag inner container. The sample was placed inside the polyethylene bag and the bag was sealed with an accompanying wire seal. The bag was then sealed inside the paint can by forcing the top securely down onto the can. Fiberglass tape was provided to assure that the lid of the can would not inadvertently be jarred loose during shipment (Fig. 10).

A franked self-addressed form was glued to the outside of the can, along with the Department of Agriculture authorization seal. Therefore, the only work required of the sampler was to scoop the soil into the polyethylene bag with the can lid, seal the unit as described above, write the Station name and APO or FPO number above the return address, and drop the can into the nearest civilian, Army, or Fleet post office mail bag.

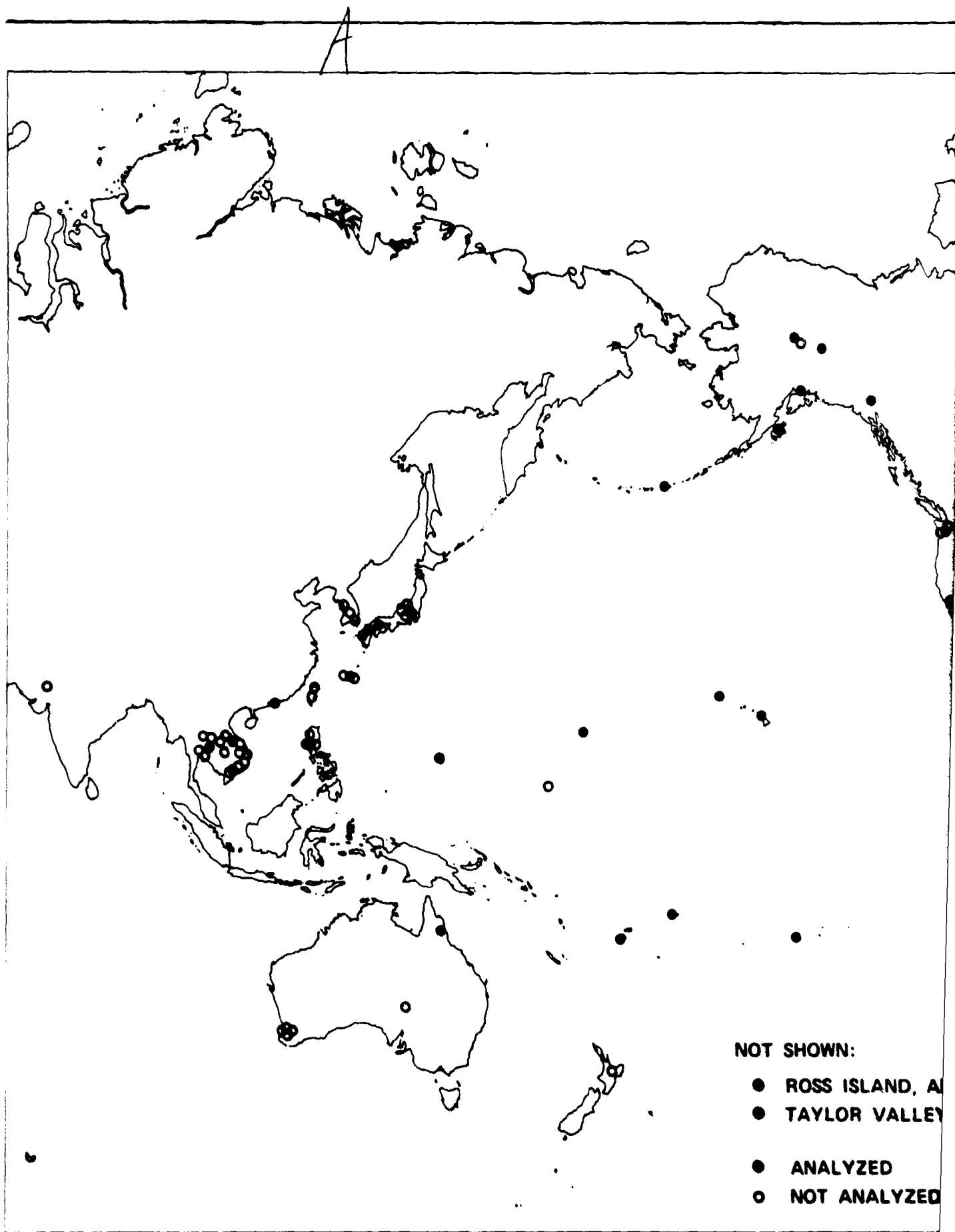
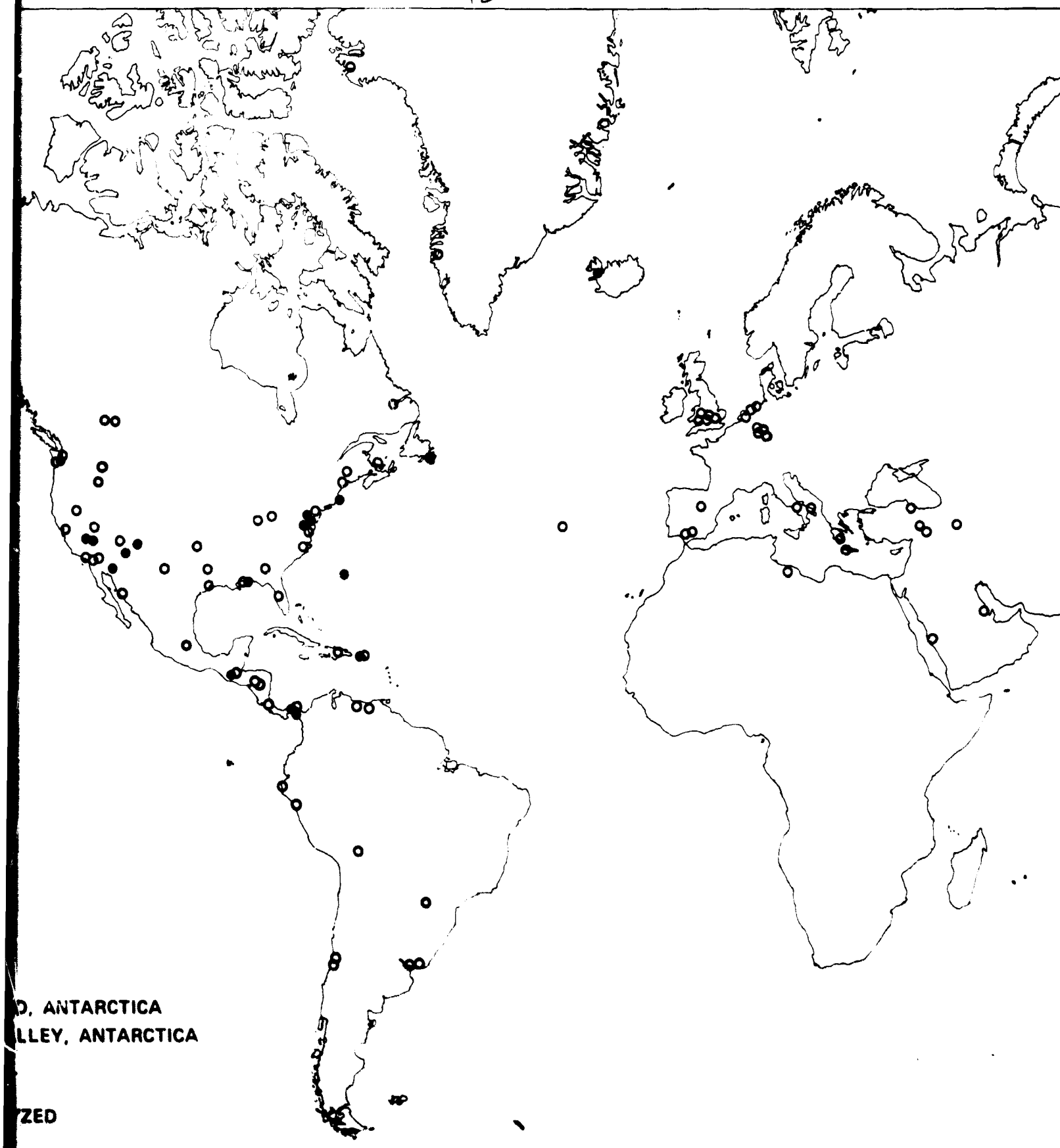


FIG. 9. Locatio

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Locations of Soil Sampling Sites.

TABLE 1. Characterization of Soil Samples.

Location	Composition (percent by weight) ^a									Ignition ^b loss, %	Density, g/cm ³	Average particle size, μ
	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	TiO ₂	MnO	CaO	MgO	K ₂ O	Na ₂ O			
Da Nang, Vietnam	80.21	7.61	8.69	0.68			0.08			3.35	2.735	20
Korat, Thailand	77.37	8.90	3.97	0.67			0.29			6.93	2.654	28
Subic Bay, Philippine Islands	39.07	29.22	15.34	1.70			0.20			13.27	2.851	14
Hong Kong	74.75	11.94	2.59	0.40	0.06	0.84	0.13	3.30	0.88	5.00	2.70	9
Naha, Okinawa	67.59	12.15	4.59	c		5.37	1.46			6.41	2.731	21
Iwotome, Japan	67.94	16.17	4.85	c		2.92	0.89			2.14	2.626	32
Atsugi, Japan	32.54	26.45	15.40	c		1.02	1.96			13.97	5.128	d
Sosobe, Japan	69.83	12.46	5.72	c		0.31	0.63			6.93	2.700	22
Agana, Guam	14.09	26.75	15.37	c		12.28	0.40			27.31	3.239	17
Fiji Island	43.99	23.01	12.23	0.93	0.14	3.76	2.98	0.27	2.33	7.63	3.03	4
Noorua, Tahiti	15.69	2.15	1.93	0.33	0.03	35.58	2.22	0.33	0.77	39.69	2.93	8
Pago Pago, Amer. Samoa	13.25	6.08	6.29	0.93	0.10	39.23	3.65	0.29	0.65	28.63	3.20	7
Wake Island	0.13	M11	0.99	--		51.12	1.23			44.54	2.780	36
Midway Island	29.99	22.14	21.37	c		2.88	0.91			16.57	3.391	15
Oahu, Hawaii	31.71	21.73	26.32	c		0.60	0.94			14.62	4.546	13
Imtisfall, Queensland, Aust.	32.81	28.32	22.69	2.85	0.13	0.75	0.55	0.05	0.15	12.06	3.08	5
Adak #1, Alaska	54.27	25.49	1.80	c		11.45	4.37			0.40	2.899	188
Adak #2, Alaska	31.09	13.79	2.30	c		2.86	0.49			44.78	2.072	22
Anchorage, Alaska	64.94	15.84	5.69	0.90		0.70	1.84			4.19	2.728	35
Kodiak, Alaska	57.06	16.39	6.66	c		1.98	1.54			11.34	2.387	10
Tanana Valley, Alaska	81.43	7.15	3.37	0.63		1.80	1.44			1.52	2.690	45
Alcan Highway (Danson Creek-Delta Junction)	56.70	14.51	6.48	0.85		7.75	3.65			7.91	2.744	8
White Horse, Yukon	68.14	13.22	3.13	0.60		5.66	1.88			3.96	2.476	20
Sea-Tac, Wash.	66.60	14.12	3.70	0.73		0.58	3.17			8.30	2.543	34
China Lake, Calif.	69.50	13.22	3.97	c		5.47	1.15			2.58	2.685	61
Sierra Nevada (Fish Creek), Calif.	54.57	18.85	10.37	c		6.71	3.20			3.00	2.796	36
Yuma, Arizona	82.07	5.80	1.30	0.28		4.84	1.55			2.75	2.646	47
Flagstaff, Arizona	54.28	18.31	10.57	c		4.33	2.44			5.38	3.274	--
Four-State Corners, U.S.	83.01	6.22	1.37	c		2.00	0.65			2.87	2.777	>25
Providence, R. I.	76.83	11.41	2.23	c		1.64	0.43			4.75	2.718	20
Harrisburg, Pa.	68.41	13.22	5.35	1.10		1.10	1.63			7.46	2.711	10
Fairfax, Va.	65.18	14.16	7.28	1.37		2.28	1.35			6.39	2.755	19
Eglin AFB, Fla.	95.18	1.94	0.31	c		0.49	0.52			1.10	2.644	>52
Guatemala City, Guatemala	4.74	20.07	7.41	c		5.45	1.15			17.99	2.796	10
Ft. Clayton, Panama	36.73	25.86	16.71	c		0.37	0.44			12.23	4.239	11
Coco Sola, Panama	44.50	24.55	10.08	c		0.21	0.99			12.38	4.500	11
Bermuda	2.11	1.75	0.79	c		50.05	0.95			42.46	2.699	26
San Juan, Puerto Rico	36.53	7.10	3.33	0.28	0.06	25.43	0.75	0.57	0.67	24.20	2.93	7

Locality	77.37	8.80	3.97	0.67	0.06	0.84	0.20	0.29	0.88	13.27	2.851	14
Korut, Thailand	39.27	29.22	15.34	1.70							2.70	9
Subic Bay, Philippine Islands	74.75	11.94	2.59	0.40					3.30	5.00	2.731	21
Hong Kong	67.59	12.15	4.59	c		5.37	1.46			6.41	2.626	32
Kobe, Okinawa	67.94	16.17	4.85	c		2.92	0.89			2.14	5.128	d
Fukushima, Japan	32.54	26.45	15.40	c		1.02	1.96			13.97	2.700	22
Atsugi, Japan	69.63	12.46	5.72	c		0.31	0.63			6.93	3.239	17
Sasebo, Japan	14.09	26.75	15.37	c		12.28	0.40		0.27	27.31	3.03	4
Apapa, Spain	43.99	23.01	12.23	0.93	0.14	3.76	2.98		0.33	7.63	2.93	8
Fiji Island	15.69	2.15	1.93	0.33	0.03	35.58	2.22		0.65	39.69	3.20	7
Norma, Tahiti	13.25	6.08	6.29	0.93	0.10	39.23	3.65			28.63	2.780	36
Papeete, Amer. Samoa	0.13	Nil	0.99	--		51.12	1.23			44.54	3.391	15
Wake Island	29.99	22.14	21.37	c		2.88	0.91			16.57	4.546	13
Midway Island	31.71	21.73	26.32	c		0.60	0.94		0.15	14.62	3.08	5
Oahu, Hawaii	32.61	28.32	22.69	2.85	0.13	0.75	0.55			12.06	2.899	188
Innisfail, Queensland, Aust.	54.27	25.49	1.80	c		11.45	4.37			0.40	2.072	22
Adak #1, Alaska	31.09	13.79	2.30	c		2.86	0.49			44.78	2.728	35
Adak #2, Alaska	64.94	15.84	5.69	0.90		0.70	1.84			4.19	2.387	10
Anchorage, Alaska	57.06	16.39	6.66	c		1.98	1.54		0.05	11.34	2.690	45
Kodiak, Alaska	81.43	7.15	3.37	-0.63		1.80	1.44			7.91	2.744	8
Tenana Valley, Alaska	56.70	14.51	6.48	0.85		7.75	3.65			3.96	2.476	20
Alcan Highway (Densen Creek - Delta Junction)	68.14	13.22	3.13	0.60		5.66	1.88			8.30	2.543	34
White Horse, Yukon	66.60	14.12	3.70	0.73		0.58	3.17			2.58	2.685	61
Sea-Tac, Wash.	69.50	13.22	3.97	c		5.47	1.15			3.00	2.796	36
China Lake, Calif.	54.57	18.85	10.37	c		6.71	3.20			2.75	2.646	47
Sierra Nevada (Fish Creek), Calif.	82.07	5.80	1.30	0.28		4.84	1.55			5.38	3.274	--
Yuma, Arizona	54.28	18.31	10.57	c		4.33	2.44			2.87	2.777	>25
Flagstaff, Arizona	83.01	6.22	1.37	c		2.00	0.65			4.75	2.718	20
Four-State Corners, U.S.	76.83	11.41	2.23	c		1.64	0.43			7.46	2.711	10
Providence, R. I.	68.41	13.22	5.35	1.10		1.10	1.63			6.39	2.735	19
Harrisburg, Pa.	65.18	14.16	7.28	1.37		2.28	1.35			1.10	2.644	>52
Fairfax, Va.	95.18	1.94	0.31	c		0.49	0.52			17.99	2.796	19
Edlin AFB, Fla.	42.74	20.07	7.41	c		5.45	1.15			12.23	4.239	11
Guatemala City, Guatemala	36.73	25.86	16.71	c		0.37	0.44			12.38	4.500	11
Ft. Clayton, Panama	44.50	24.55	10.08	c		0.21	0.99			42.46	2.699	26
Colon, Panama	2.11	1.75	0.79	c		50.05	0.95		0.57	24.20	2.93	7
Bermuda	36.53	7.10	3.33	0.28	0.08	25.43	0.75		1.10	63.88	1.34	19
Ramir AFB, Puerto Rico	15.73	9.79	3.49	0.48	0.06	1.39	1.19			15.99	3.368	6
Argentina, Newfoundland	31.34	23.86	15.25	c		3.89	1.27		1.41	8.34	2.97	8
Reykjavik, Iceland	69.77	7.40	4.99	0.47	0.14	4.42	0.48		1.83	2.86	3.09	12
Heyford, England	44.17	14.36	13.69	3.55	0.22	9.27	8.61		2.25	2.11	2.98	10
Boss Island, Antarctica	60.77	12.96	7.08	1.08	0.12	5.61	4.74					
Taylor Valley, Antarctica												

NOTE: Absence of data in composition section does not mean oxides were not present; depends on testing technique.

a All metals reported as oxides.

b Ignition loss: 1 hour at 1292°F.

c Any minor amount of TiO₂ would be included in the Al₂O₃ value.

d Porosity too high, out of range. Particles are large fused agglomerates which crush to micron size particles.

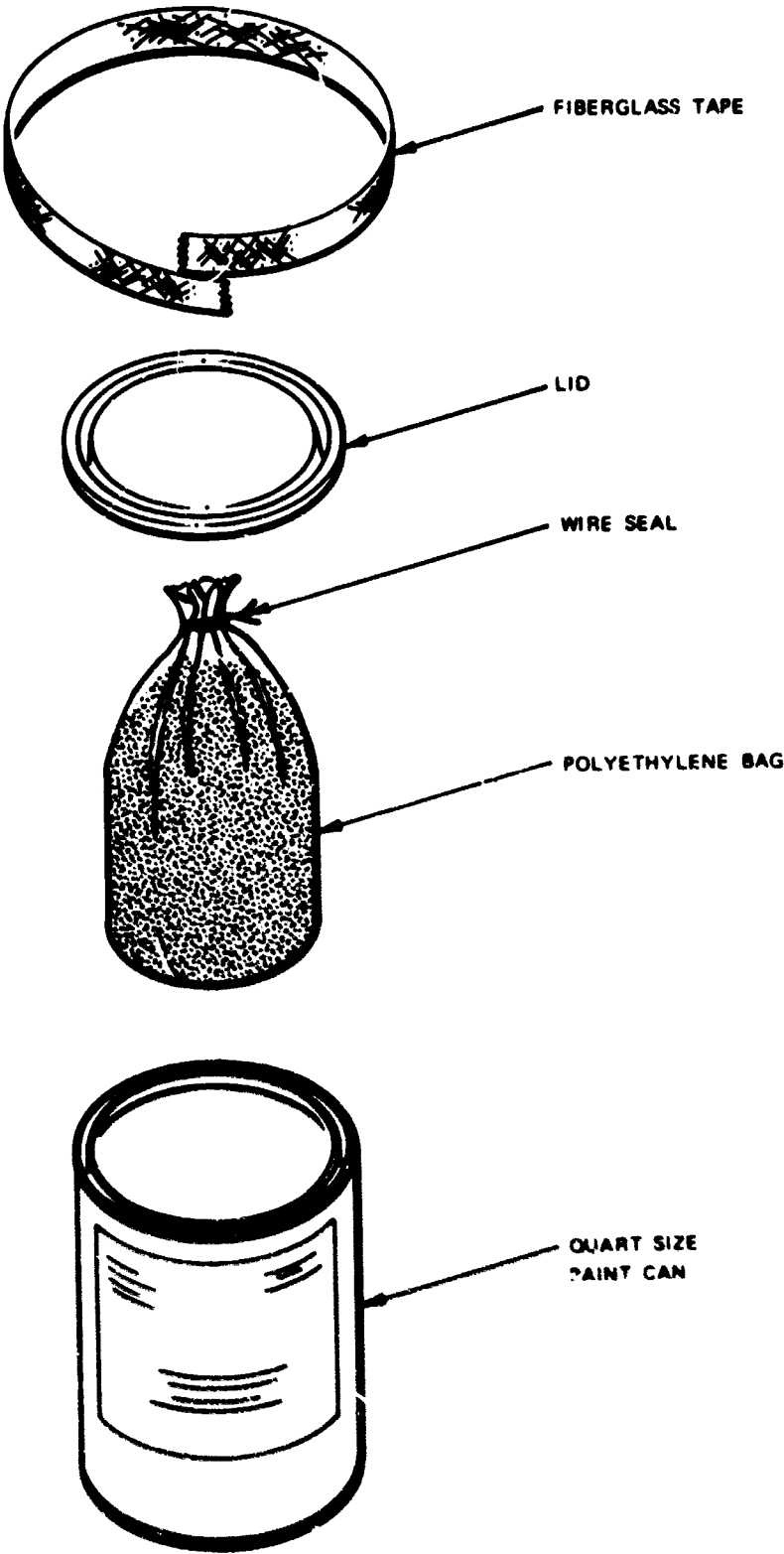


FIG. 10. Container for Soil Samples.

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Before starting the project, the Department of Agriculture was contacted so that no health or quarantine regulations would be broken by the shipment of foreign soil into the Continental United States. For the continuing project, NWC was granted authorization number "Ltr 10-25-67 ZAB, Schafer, Nav. Env. Center, Soil Samples for Release Without Treatment."

ANALYSES TECHNIQUES

After the sample had been subjected to the heat sterilization cycle (Appendix B), it was given a preliminary separation treatment. The pieces of thermally-altered plastic, from the polyethylene shipping bag, were separated manually from the soil sample. Then, a U. S. Standard No. 18 sieve (1,000 micron openings) was used to remove any coarse matter, such as pebbles, roots, twigs, or other material, which was considered as not being an actual part of the soil sample.

Next, the samples were subjected to quantitative analysis. The compositions of 33 samples were determined by wet chemical analysis and 10 samples were primarily analyzed by atomic absorption spectroscopy (AA).

A semiquantitative spectrographic analysis was run on each soil sample prior to the wet chemical analysis. This indicated the approximate concentrations of the constituents in the sample. Then, a quantitative wet-chemical analysis scheme was employed to determine the percentage composition. A complete description of the procedures used in the wet chemical analyses of soil samples is given in Appendix C.

The samples from 10 locations (those requiring nine columns in the composition section in Table 1) were analyzed by a combination of techniques. Ignition losses and silica determination were performed by standard conventional methods (Appendix C). The remainder of the analysis was by AA. First, a 0.1 gram sample was fused with lithium metaborate, the melt taken up with dilute HCl into a 200-ml volumetric flask. All subsequent analyses were performed on this stock solution or dilutions to bring the absorbance down to a working range. All the analyses were by the method of standard additions.

The density values were determined by the use of the Beckman Model 930 Air Comparison Pycnometer and the average particle size by the Fisher Sub-Sieve Sizer.

The Beckman IR 10 infrared spectrophotometer (IR) was used in a limited capacity during the initial testing period. The procedure is rapid, economical, and was proven to be a reliable technique (Ref. 4 and 5). It can be used to give accurate supplementary information that can complement the data obtained by the other techniques. This will be advantageous when numerous samples are being analyzed.

Differential thermal analysis (DTA) thermal patterns were obtained on some soil samples. The Deltatherm DTA System (D2000) was used to make the runs. All runs were made under essentially the same operational conditions so that qualitative comparisons are possible. The thermal patterns are presented in Appendix D.

RESULTS

CHEMICAL ANALYSIS

The wet chemical analysis scheme revealed that the soil sample from Eglin Air Force Base, Florida, was the only one that gave values that were essentially in agreement with the requirements in the specification MIL-STD-810B. The soil sample contained 95 percent silica (SiO_2), whereas the specification called for 97-99 percent silica. The minor constituents were within specification limits.

The quantitative data (Table 1) showed that there were wide variations in the percentage compositions of the three principal constituents. The silica content ranged from 0.13 to 95 percent, the alumina content from Nil to 29 percent and the iron oxide content from 0.3 to 26 percent.

The data showed the existence of significant variations within a relatively small area of interest. It has been shown that although the chemical analysis was essentially the same for two sites approximately 435 miles apart, the values were drastically different for a site approximately halfway between these two. The quantitative chemical analysis on the soil samples from Yuma, Arizona, and Four State Corners (junction of Utah, Colorado, Arizona, New Mexico) gave about the same percentage values: ignition loss 2.8, silica (SiO_2) 82, alumina (Al_2O_3) 6, and iron oxide (Fe_2O_3) 1.3 (Note: all metals reported as oxides). However, the analysis on a soil sample from Flagstaff, Arizona, (midway), was entirely different. The ignition loss was twice as high, the silica content about 34 percent lower, the alumina content about three times greater and the iron oxide content about eight times greater.

During the initial testing period, partial preliminary analyses were performed on a few soil samples from two widely separated areas. This work utilized the infrared spectrophotometer. The spectra indicated that the principle constituent in the samples from the southern Californian area was silica (SiO_2) and in the samples from the southeast Asian area it was China Clay ($\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2 \cdot 2\text{H}_2\text{O}$).

To have a better understanding of the significance attached to some of the major constituents, alone and in various combinations, some of their respective properties are reviewed to indicate some possible

physical/chemical relationships and also what courses of action can be initiated or aggravated by certain combinations under different environmental conditions.

Alumina (Al_2O_3) and its monohydrate have values of about 9 and 7 on the Mohs' Scale of Hardness (diamond has a top value of 10). It is apparent that they can cause serious erosive and cutting damage to metallic and plastic structures. However, when the di- and tri-hydrates are formed, the hardness value is lowered and it ranges from "soft" to 3.5. Although Al_2O_3 is generally found as a constituent of clays, there were evidences where this was different. For example, the DTA thermal patterns on soil samples from Adak No. 1 and Anchorage did not show any endothermic action in the 500-600°C region. This would indicate the presence of Kaolinite, the main constituent of China Clay. However, the chemical analysis showed that the aluminum values, reported as an oxide, were 25 and 16%. If most of the water of hydration was removed, the end product could cause serious damage by erosion.

When silica (main constituent of sand) becomes hydrated, its hardness value only decreases from 7 to 5.5-6.5. Therefore, both states can still cause extensive damage to a variety of materials. The magnitude of the problem is well known; for example, at NWC it has been witnessed that painted surfaces have been sandblasted clean within a 2 minute period by a high velocity sandstorm.

The proper combination of alumina, silica, and water will produce a soft, clay-like material with a hardness of about 2. Even though this material would not be erosive, it could be corrosive because of the presence of moisture. Deterioration due to corrosion could be a relatively long-term affair. A serious short-term effect may develop if red iron oxide (Jewelers' rouge) is present within the China clay. The soft material will act as a vehicle for the oxide, forming an erosive compound. This compound has a cutting and polishing effect on any metallic surface, and its effect will even be more severe if additional water is present. Should this combination get into truck brake drums or into aircraft engines, for example, then a serious erosive situation is possible and it can cause dramatic results within a short time period. The brakes may cease to operate properly or the brake drums may even be eroded enough to fall off, and the aircraft engines may fail to operate.

Some combinations may not cause erosive damage during the wet season, but problems may occur during the seasonal dry spells. During these dry periods, for example, the wet clay-like material develops into a fine, dust-like substance which is extremely irritating, erosive, and contaminating (such as the stopping up of various filter systems). Therefore, the erosive and corrosive mechanisms may be active simultaneously or they may alternate, depending on the prevailing climatological conditions.

IGNITION LOSS

The loss in weight on ignition, to 1292°F, would indicate the quantity of organic material, occluded water, or perhaps calcite which is present. It may indicate the level of sampling, surface or subsurface. This is important since it is the so-called subsoil, that portion beneath the humus layer, which causes seemingly the most damage to jet aircraft engines, military vehicles, and ordnance items. In many areas, this so-called subsoil is or becomes the topsoil which comes into active contact with military hardware at airfields, on roads and in open terrain.

A relatively large ignition-loss value, 13-64 percent, may give a clue as to the generalized type of climate at the sampling site, e.g., one which is conducive to the establishment of heavy vegetation or the presence of other organic matter. High values were obtained on samples originating from the Philippine Islands, Adak #2, Alaska, Argentina, and the Central American region. The high ignition-loss values for the samples from Adak #2, Alaska and Guatemala City are probably due to the presence of organic matter. This is indicated in their DTA thermal patterns, i.e., exothermic action in the 392-842°F (200-450°C) region. These percentage values are in addition to the quantity of volatiles and solid organic matter lost during the preliminary sterilization treatment, which was 350-400°F for 16-20 hours.

A high ignition-loss value may also be due to the decomposition of inorganic matter. High values were found on the samples from Wake Island, Bermuda, Pago Pago, and Tahiti. The DTA thermal patterns on the samples from Wake Island and Bermuda show only a small amount of organic decomposition but they do show a large amount of decomposition in the 1157-1472°F (625-800°C) region. This is evidently due to the presence of calcite, the most common form of calcium carbonate (CaCO_3). Calcite decomposes in the temperature range of 1112-1490°F (600-810°C), which would be affected by the ignition temperature of 1292°F (700°C). This is substantiated by the high calcium content (~50%) found by chemical analysis. Calcium carbonate decomposes in the following manner: $\text{CaCO}_3 \rightarrow \text{CaO} + \text{CO}_2$ (carbon dioxide gas).

DENSITY

Currently, the density values are of academic interest only. However, there is an apparent relationship between the amount of iron oxide present and the density of the soil sample. Therefore, this density/composition relationship can be utilized as a qualitative checkpoint on the amount of minerals or mineral compounds present in the soil sample.

AVERAGE PARTICLE SIZE

An average particle value has been reported for each sample. In Table 1, 88 percent of the samples had an average particle size of less than 40 microns, 10 percent were between 40-74 microns, and 2 percent were greater than 74 microns. The average value represents a point on a typical bell-shaped distribution curve, wherein the particle size-range can be rather large. However, it is still a significant value and can be used to categorize a number of samples. It is realistic, providing it represents true particle size and not agglomerates.

These values were determined by the Fisher Sub-Sieve Sizer (FSSS) and are obtained by measuring the pressure due to resistance to air flow through the material. It is possible to have a mixture of parent particles and agglomerates. Some or all of the agglomerates may be broken up and separated, depending on the degree of compactness and the nature of the airflow. This phenomenon is more pronounced if the Ro-Tap method is employed (mechanically vibrating a series of screens with different openings). This method is not satisfactory for agglomerate soil samples because the retained volumes are dependent on the time of operation of the Ro-Tap device, and not on the parent values. At Da Nang, for example, the soil agglomerates into large masses during the wet season. It then dries out into chunk-like pieces. When this dirt is vibrated on the Ro-Tap, it is possible to obtain any sample distribution the operator desires, depending on the vibration time. The agglomerate is basically made up of particles in the 10-20 micron range.

It has been reported in Ref. 6 that the majority of the airborne dust particles (collected at the air inlet of Army tanks) were less than 40 microns. It indicated also that other sources reported the dust plumes, caused by vehicles, were almost all less than 74 microns. It is possible that appreciable quantities of dust can be produced almost anywhere in the world providing the soil moisture content is low and the surface agglomeration of the particles is not excessive.

The use of helicopters has increased considerably during the past few years, especially in the Vietnam theater of operations. The extremely high air velocities generated by a helicopter can cause the movement of particles >200 microns, causing erosion damage to the helicopters as well as to nearby equipment on the ground.

SUMMARY

It has been demonstrated that soils of the world can be sampled in an easy manner and that, even though there are anomalies in samples no more than a few miles distant, world dirt as a whole can be easily classified. The 43 soil samples analyzed so far would indicate that the present

"standard" understanding of the world's soil composition is somewhat in error. Of the 43 soil samples analyzed, only one (Eglin Air Force Base) gave values that were essentially in agreement with the Sand and Dust Specification MIL-STD-810B.

The total of 264 dirt samples already on hand at NWC from the worldwide sources definitely indicate that more destructive components should be recognized. The universality of the Al_2O_3 and Fe_2O_3 as seen in the analysis of the 43 samples and the telltale red color found on visual inspection of the remainder of the samples would seem to indicate that those oxides of iron and aluminum are of worldwide concern. Since the inception of this project, because of Vietnam failure reports, one author (Howard Schafer) has, during world travel, been impressed by the predominance of the red soil coloration. Even in the Continental United States there are large areas where red soil is predominant. Where the soil is red, the oxides of iron and aluminum can be in notable concentrations.

It also must be stated that during this type of soil sampling many anomalies may be missed. However, even one sample from Thailand, Vietnam, and the Philippines, for example, will indicate the general situation for that part of the world. If the analysis of the three samples are similar, as was the case for the first two, then the generalization can take on more meaning. It is stipulated that the more numerous the samples, the more accurately the minute details will be known. After studying the dirt samples from all parts of the world, except the African continent, it would seem that though scientific differences are quite significant, engineering differences may not be significant. Based on the results of the present work, it may be relatively simple to determine a "worldwide standard dirt" for simulating worldwide environmental conditions.

RECOMMENDATIONS

It is recommended that this work be continued to obtain additional soil samples from areas not yet covered and to conduct additional analysis as required to provide a comprehensive data basis for revision of the present specification.

It is also recommended that the problem of soil induced corrosion be further studied. It was only after the authors were well into the mechanical implications as indicated in this report that the corrosion potential of "gummy" clays and other "soft" dirt compounds was realized.

In addition, all clays break down exothermally to alumina at temperatures of approximately 2000°F (Ref. 7). The energy from the reaction is similar to that given off by a Thermit grenade. Since temperatures above 2000°F are present in all jet and internal combustion engines, this phenomena could be the cause of the reduced life of aircraft engines at unimproved airfields due to the following:

1. When the clays break down to alumina they give off exothermic heat, possibly causing high temperature corrosion.
2. Extremely small particles are formed.
3. The particles are extremely hard and therefore very erosive.

Appendix A

ADDITIONAL SOIL SAMPLES AVAILABLE FOR ANALYSIS AT NWC

Nha Trang AB, Vietnam (2)	John Hay Air Base, Baguio, Luzon
Bien Hoa AB, Vietnam (3)	Cubi Point, Philippine Islands
Cam Ranh Bay, Vietnam	Sangley Point, Rep. of Philippines
Da Nang AB, Vietnam (2)	Naha Air Base, Okinawa (4)
Tan Son Nhut AB, Vietnam (2)	Kadena Air Base, Okinawa (5)
Phu Cat AB, Vietnam (2)	Onna Point Admin Annex, Okinawa
Dong Ha, Vietnam (YD 246-598) (2)	Osan Air Base, Korea (4)
Da Nang, Vietnam (AT 9836-7244)	Kunsan Air Base, Korea (2)
Chu Lai, Vietnam	Udonbu, Korea
XD 852-419, Map 6342-IV, Vietnam	Seoul, Korea
AT 955-805, Map 6641-III, Series L7014, Vietnam (2)	Taipei, Air Station, Taiwan (2)
AT 955-805, Beach Area, Vietnam	Shu Lin Kou Air Station, Taiwan
BT 005-738, Map 6641-IVs, L8020, Vietnam	Ching Chuan Kang Air Base, Taiwan (2)
BT 005-738, Bomb Dump, Vietnam	Fukuoka, Japan
XD 845-455, Hill 950, Khe Sanh, Vietnam	Tachikawa Air Base, Japan (3)
Bing Thuy AB, Vietnam	Misawa Air Base, Japan
Nakhon Phanom Airport, Thailand (2)	Yokota Air Base, Japan (2)
Udon Airfield, Thailand (2)	Itazuke Air Base, Japan
U-Tapao Airfield, Thailand (2)	Wakkanai Air Station, Japan
Takhli Air Base, Thailand (2)	Fuchu Air Station, Japan (2)
Ubon, Airfield, Thailand	Camp Drake, Japan
Kabinburi, Thailand	Pago Pago, American Samoa
Pak Chung, Thailand	Eniwetok, Marshall Islands
Don Maung Airport, Thailand	Hickam Air Force Base, Hawaii (3)
Clark Air Base, Philippine Islands (9)	Barbers Point, Oahu, Hawaii
Mactan Isle Airfield, Philippine Islands (2)	Bellows Air Force Base, Oahu, Hawaii
	Kunia Facility, Hawaii
	Kaneohe Bay, Hawaii
	Ft. Ruger, Hawaii
	South Australia

Grantsville, Australia	White Sands, New Mexico
Woomera, Lake Bed, South Central Australia (2)	Indian Head, Maryland
Perth, West Australia (Nature Reserve) (3)	Sanford, Florida
Perth, West Australia (Beach Sand ~5-10 miles north of Perth-Indian Ocean Coastline)	Pensacola, Florida
Perth, West Australia (Natural growth ~10 miles north of Perth)	Seattle, Washington
Perth, West Australia (~50 miles south of Perth)	Yorktown, Virginia
Perth, West Australia (~20 miles south of Perth)	Charleston, Virginia
Perth, West Australia (~25 miles south of Perth)	Dam Neck, Virginia
Perth, West Australia (~30-40 miles south of Perth)	USNAD Earle, Colts Neck, New Jersey
Bibra Lake, West Australia (~40 miles east of Perth)	Brunswick, Maine
Perth, West Australia (~5 miles north of Perth, inland)	Marble Mountain Air Facility, Maine
Wairakei, New Zealand	Cherry Point, North Carolina
Rotorua, New Zealand	Crane, Indiana
Eielson AFB, Alaska	McAlester, Oklahoma
Bremerton, Washington	Orrville, Ohio
Hawthorne, Nevada (2)	NAS, Albany, Georgia
Alturas, California	Panama, CZ
San Diego, California	Albrook Air Force Base, Balboa, CZ
El Centro, California	San Juan, Puerto Rico (2)
Seal Beach, California	Sondrestrom Air Base, Greenland (2)
Concord, California	Thule Air Base, Greenland
El Toro, California	Goose Air Base, Canada (2)
Yuma, Arizona (No. 2)	Prince Edward Island, Canada (beach sand)
Bottom Grand Canyon, Arizona	Lajes Field, Azores
NAS, Corpus Christi, Texas	Chicksands, England
Dallas, Texas	West Ruislip, England
	Croughton, England
	St. Morgan, England
	High Wycombe Air Station, Buckinghamshire, England (2)

Upper Heyford, England	Teheran, Iran
Wethersfield, England	Jidda Airfield, Saudi Arabia
RAF, Alconbury, Huntingdonshire, England (2)	Dhahran Airfield, Saudi Arabia
Bentwaters, England	Wheeler Air Base, Libya (2)
Mildenhall, England	Pes Havar Air Station, Pakistan
Darmstadt, Germany	APC 96319 CCK
Wiesbaden Air Base, Germany (3)	Costa Rica (2)
Hahn Air Base, Germany (3)	Jicaró Galán, Honduras
Bitburg Air Base, Germany	Tegucigalpa, Honduras
Sembach Air Base, Germany (2)	Anaco, Venezuela
Rhein-Main Air Base, Germany (2)	Caracas, Venezuela
Ramstein Air Base, Germany (2)	Chimbote, Peru
Det 2, 36th TFW, APC NY 09292 (Europe)	Cabo Blanco, Peru
Seville, Spain	Guaymas, Mexico
Torrejón Air Base, Spain (5)	Santiago, Chile
Zaragoza Air Base, Spain	La Quinta, Chile
Soesterberg, Netherlands	Montevideo, Uruguay
Camp New Amsterdam Air Base, Netherlands	Las Flores, Uruguay
Brussels, Belgium	Guatemala (2)
Brindisi, Italy	Jasper, Alberta, Canada
Aviano Air Base, Italy (2)	Edmonton, Alberta, Canada
Athens Airport, Greece	Missoula, Montana
Iraklion Air Station, Crete, Greece (2)	Challis, Idaho
Samsun Air Station, Turkey	Santiago, Dominican Republic
Diyarbakir Air Station, Turkey	Santa Domingo, Dominican Republic
Malatya, Turkey	La Paz, Bolivia
	Mexico City, Mexico
	Puerto Pres. Stroessner, Paraguay

Appendix B

STERILIZATION OF SOIL SAMPLES

During the Sand and Dust Survey Program, soil samples were received from various areas of the world. It was necessary to consider the possibility that the samples could contaminate areas and personnel. To avoid this possibility it was decided that all soil samples, whether coming from within or outside the continental limits of the United States, would be subjected to a rigorous sterilization treatment before they would be handled in any manner.

The temperature-time limits were established after a literature search was made. Burrows¹ has indicated that, for the sterilization of glassware and instruments by dry heat, the temperature should be between 170 and 180°C (338-356°F) and it should be maintained for a period of not less than 2 hours. Sykes² has reported the destruction times for various bacterial spores that have been subjected to different temperatures (dry heat). At 170°C (338°F), it required 15-60 minutes for soil spores to be destroyed and 10-15 minutes for the *C. botulinum* organisms. At 180°C (356°F), the *C. botulinum* organisms were destroyed in 5-10 minutes and the *B. anthracis* in 3 minutes.

The sterilization procedure utilized available equipment and was carried out at an operational test site. An NWC standard 8-inch-diameter cook-off oven was used. It would accommodate six containers simultaneously. To establish the correct operational procedure, a number of test samples were prepared, using the same type of container and confinement as would be used for the incoming soil samples. Sand was used for the test sample, and about 200 ml of water was added to each batch so that the final sample would be extremely moist and it would be representative of soil samples containing the most moisture. The usual amount of soil, inside a secured plastic bag, was placed inside the metal cans and the lids were secured. Some of the lids were provided with a 1/8-inch vent hole. The temperature in the oven was set for 400°F and the containers remained in the oven overnight. When they were examined the following morning, the containers with the vent hole were still intact, while the others, with no vent, blew open and the soil sample was scattered in and outside the oven. Next, thermocouples were utilized to determine the dwell time before the temperature in the center of the sample reached 350°F. When the container was placed within the preheated 400°F oven, the temperature within the center of the sample increased to about 200°F, leveled off until the moisture evaporated, then quickly rose up to the oven temperature. It required about 5.6 hours

¹Burrows, William. Textbook of Microbiology, 18th Edition. Saunders, Philadelphia, 1963.

²Sykes, G. Disinfection and Sterilization. D. Van Nostrand, New York, 1958. Pp. 96.

for the center of the sample to attain the temperature of 350°F. The inner plastic bag did not burn, but melted and congealed into irregular, globular masses. Since the thermally altered plastic penetrated only a minute distance into the soil sample, a sufficient amount of the indigenous sample remained for the required testing.

For the incoming soil samples, the operational procedure did not require the placing of thermocouples within the container since a sufficient number of runs had already been made to establish the thermal profile within the soil sample. Only a vent hole was made in the lid of the container before the container was placed within the oven.

An additional precautionary step was taken for the sterilization of soil samples originating outside the continental limits of the United States. The container and a nail were placed inside an oversized plastic bag and the bag was closed and secured by a piece of tie-wire. The head of the nail had been padded with tape to prevent it from causing a hole in the plastic bag. The point of the nail was positioned against the lid and the padded end was struck with a plastic hammer. When this technique was used, the bag remained intact. Any probable pathogenic organisms were contained within the bag and could not, therefore, infect personnel or contaminate the environment. The entire assembly (container inside the bag) was placed in the preheated oven for the sterilization treatment. During the heating cycle, the bag melted, but did not cause any damage to the oven. All containers were properly marked with identification numbers or letters before they were placed in the oven. If possible, the paper shipping labels were removed before the heat treatment and were replaced after the containers had returned to ambient temperature.

The dry heat method was recommended and employed because it was convenient, economical and effective. The temperature within the oven was maintained at 350-400°F and the sample remained in the oven overnight, giving a total heating time of 16-20 hours. The container and contents can be sterilized at the same time. This simplifies the procedure and the can and contents can be safely handled, stored or discarded.

Appendix C

CHEMICAL ANALYSES OF SOIL SAMPLES

The following procedures were used in the quantitative wet chemical analyses for the following constituents: moisture loss, ignition loss, SiO_2 , Fe_2O_3 , Al_2O_3 , TiO_2 , CaO , and MgO .

APPARATUS

1. Analytical Balance
2. Beckman DB Spectrophotometer
3. Mortar and Pestle
4. 100-Mesh Screen
5. Hot Plate
6. Muffle Furnace
7. Meker Burner
8. Desiccator
9. Volumetric Flasks
10. Pipettes
11. Platinum and Porcelain Crucibles

REAGENTS

1. Sodium Carbonate, anhydrous
2. Ammonium Chloride, granular
3. Zinc, 20-mesh
4. Hydrochloric Acid, CP
5. Hydrochloric Acid, 50%
6. Hydrochloric Acid, 1%
7. Perchloric Acid, 70% CP
8. Hydrofluoric Acid, 48% CP
9. Sulfuric Acid, CP
10. Sulfuric Acid, 50%
11. Ammonium Hydroxide, 50%
12. Bromine Water Solution, saturated

13. Ammonium Nitrate, 2.5% aqueous
14. Mercuric Chloride Solution, saturated
15. Hydrogen Peroxide, 3% aqueous
16. Ammonium Oxalate, 5% aqueous
17. Ammonium Oxalate Wash Solution, 0.1% aqueous
18. Diammonium Hydrogen Phosphate, 10% aqueous
19. Sulfuric-Phosphoric Acid Solution, 150 ml of H_2SO_4 + 150 ml H_3PO_4 (85%) to 700 ml H_2O
20. Sodium Diphenylamine Sulfonate Indicator, 0.2% aqueous
21. Methyl Red Indicator, 0.2 g in 100 ml of ethanol
22. Standard Titanium Solution (1 ml = 0.1 mg TiO_2). Dissolve 0.0500 g of dry TiO_2 in 50 ml of 1:1 H_2SO_4 . Evaporate to dense white fumes. Cool and dilute to 500 ml in volumetric flask.
23. Reagent Blank, 5 ml of 3% H_2O_2 and 10 ml of 1:1 H_2SO_4 diluted to 100 ml with H_2O in volumetric flask.
24. Standard Potassium Dichromate Solution (0.1 N). Dissolve 4.9035 g of the $\text{K}_2\text{Cr}_2\text{O}_7$ in water and dilute to 1 liter in a volumetric flask. Standardize against iron wire (purity known) using procedure No. 5 of this report.

PROCEDURE

1. Sampling - Grind the sample in a mortar until it all passes through a 100-mesh screen. Thoroughly mix the sample before weighing.
2. Moisture Loss - Weigh a 1-gram sample (100-mesh soil) in a platinum crucible to the nearest 0.1 milligram. Dry the sample in an oven overnight at 110°C . Cool in a desiccator and weigh.
3. Ignition Loss - Place the platinum crucible from the determination of the moisture loss in an electric furnace. Raise the temperature to 700°C and hold for 1 hour. Cool in a desiccator and weigh. This loss in weight represents the organic matter content as well as any combined water in the soil.
4. SiO_2 - Add to the residue from the determination of ignition loss five times its weight of pure anhydrous sodium carbonate. Mix the contents thoroughly, cover the crucible and heat at low redness until fusion becomes free of ebullition. Apply full heat of burner for 10 minutes. Cool the crucible and detach the melt by tapping the bottom of the crucible on a smooth hard surface. Place the melt in a covered 400 ml

beaker. Add 50 ml of 50% hydrochloric acid to the beaker and 20 ml to the crucible. Scrub the crucible and lid thoroughly with a rubber policeman and add the contents and washings to the beaker.

Digest on the hot plate until the melt has disintegrated. Remove from the hot plate and cautiously add 30 ml of perchloric acid to the beaker. Evaporate the solution to copious white fumes, taking care not to go to dryness. There will be considerable bumping of the solution, so acquire a technique to prevent this from occurring. This was accomplished by constant mechanical agitation of the solution which prevented superheating of the silica.

Cool and wash down the cover glass and sides of the beaker with water. Add 10 ml of hydrochloric acid, dilute to about 150 ml with water and bring to a boil. Filter through No. 41H paper, saving the filtrate and washings for the R_2O_3 determination. Police out the precipitate using a hot 1% hydrochloric acid wash solution. Continue washing the filter paper until the yellow color is discharged. Finish the washing with hot water until the washings are neutral to litmus paper.

Place the filter paper in the original platinum crucible; char and consume the paper at low heat. Complete the ignition of the precipitate at $900 - 1000^{\circ}\text{C}$. Cool in a desiccator and weigh. Add sulfuric acid to cover the silica precipitate and cautiously add hydrofluoric acid until the precipitate dissolves. Set on the hot plate and evaporate to dryness. Re-ignite the crucible to 1000°C , cool in a desiccator and weigh. The difference in weight is the silica present in the sample, which was volatilized.

The residue left in the crucible is fused with 1/2 gram of anhydrous sodium carbonate as above. The melt as well as the washings are added to the filtrate from the silica determination. Evaporate the filtrate to about 50 ml and transfer quantitatively to a 100 ml volumetric flask.

5. Fe_2O_3 - Pipette 50 ml from the flask into a 400 ml beaker. The remaining 50 ml will be used in the determinations of Al_2O_3 and TiO_2 . Add 100 ml of water and enough bromine water to impart a reddish color to the solution. Bring to a boil and boil for 10 minutes insuring complete oxidation of the solution. Cool slightly by washing down the cover glass and sides of the beaker with water. Add 10 grams of ammonium chloride and 50% ammonium hydroxide until precipitation is complete (pH 8). Bring to a boil and filter through No. 41H paper, saving filtrate and washings for CaO determination. Wash beaker twice and precipitate three times with hot 2.5% ammonium nitrate solution.

Place the original beaker under the funnel and dissolve the precipitate with 50 ml of hot 50% hydrochloric acid. Wash the filter paper several times with hot 1% hydrochloric acid. Discard the filter paper and bring the solution to a boil. Remove from the hot plate and cautiously

add zinc metal until the yellow color is discharged. Filter at once through No. 41H paper, set with 1/2 teaspoonful of zinc metal in cone, into a clean 400 ml beaker. Wash the beaker and the filter paper several times with water. Discard the filter paper and cool the beaker to room temperature.

While stirring the solution add 10 ml of mercuric chloride followed by 15 ml of sulfuric-phosphoric acid solution. Titrate the solution with standard 0.1 N potassium dichromate solution, using 5 drops of sodium diphenylamine sulfonate as indicator. Continue the titration until the first tinge of purple or violet-blue color appears and lasts for at least 30 seconds.

6. Al_2O_3 - Transfer the remaining 50 ml (volumetric flask) from the silica determination into a 400 ml beaker. Proceed as in the Fe_2O_3 determination up to and including the first filtering step. Catch the filtrate and washings in the same beaker used in the Fe_2O_3 determination. Police out the precipitate thoroughly and wash several times with hot 2.5% ammonium nitrate.

Evaporate the combined filtrate to about 300 ml and save for the CaO determination. Place the filter paper in a tared crucible. Char the paper at dull red heat and then ignite the precipitate at 900 - 1000°C for 1 hour. Desiccate and weigh. This weight represents combined Fe_2O_3 + Al_2O_3 and TiO_2 (R_2O_3 group).

7. TiO_2 - Transfer the combined residue (R_2O_3 group) quantitatively to a 50 ml beaker. Add 10 ml of hydrochloric acid and 10 ml of 50% sulfuric acid and evaporate to dense white fumes. Cool, wash down cover glass and sides of beaker with water, and transfer quantitatively to a 100 ml volumetric flask. Moderately heat the flask until a clear solution is obtained. If after 1 hour the solution remains cloudy, filter into another flask.

To the cooled flask add 5 ml of 3% hydrogen peroxide solution and dilute to volume. Read the absorbance at 410 m μ against a reagent blank. Read the weight of TiO_2 from the calibration curve prepared as follows:

Pipette 2, 4, 6, 8, and 10 ml aliquots of the standard titanium solution into 100 ml volumetric flasks and partially fill with water. To each flask add 5 ml of 3% hydrogen peroxide solution and dilute to volume. Read the absorbance of each solution at 410 m μ against a reagent blank. Plot the absorbance against the mg of TiO_2 and draw the curve. Read the soil sample absorbance from the curve to determine the mg of TiO_2 present.

8. CaO - The reserved filtrate from the R_2O_3 determination is made acidic with 50% hydrochloric acid. Use methyl red as the indicator. Heat to boil and add 30 ml of 5% ammonium oxalate solution. While

stirring add 50% ammonium hydroxide until the color changes to yellow, then 5 drops more. Let the solution stand for 1 hour (no longer) with occasional stirring during the first 30 minutes.

Filter through No. 40 paper into a 600 ml beaker and wash several times with cold ammonium oxalate wash solution. Save the filtrate and washings for the MgO determination. Place the filter paper in a tared crucible and char the paper without inflaming. Ignite the covered crucible at 1000°C for 1 hour. Cool in a desiccator and weigh.

9. MgO - Acidify the filtrate from the CaO determination with hydrochloric acid and evaporate to 300 ml. Add 30 ml of 10% diammonium hydrogen phosphate and cool to room temperature. Add 50% ammonium hydroxide dropwise, while stirring constantly, until the precipitate starts forming. Set aside in a cool place overnight.

Filter through No. 40 paper, police out the precipitate and wash several times with ammonium nitrate wash solution. Place the precipitate in a tared crucible and char the paper slowly. Carefully burn the resulting carbon and then ignite the precipitate at 1000°C for 1 hour. Cool in a desiccator and weigh.

CALCULATIONS

1. Moisture Loss

$$\frac{\text{Loss in weight, grams}}{\text{Oven-dry weight, grams}} \times 100 = \% \text{ Moisture loss}$$

2. Ignition Loss

$$\frac{\text{Loss in weight, grams}}{\text{Oven-dry weight, grams}} \times 100 = \% \text{ Ignition loss}$$

3. SiO₂

$$\frac{\text{Loss in weight, grams}}{\text{Oven-dry weight, grams}} \times 100 = \% \text{ SiO}_2$$

4. Fe₂O₃

$$\frac{A \times B \times 2 \times 0.07985}{\text{Oven-dry weight, grams}} \times 100 = \% \text{ Fe}_2\text{O}_3$$

A = Volume of K₂Cr₂O₇

B = Normality of K₂Cr₂O₇

5. TiO_2

$$\frac{C \times 2}{\text{Oven-dry weight, grams}} \times 100 = \% \text{TiO}_2$$

C = grams of TiO_2 from calibration curve

6. Al_2O_3

$$\frac{D \times 2}{\text{Oven-dry weight, grams}} \times 100 = \% \text{Al}_2\text{O}_3$$

D = weight of R_2O_3 in grams minus the weights of Fe_2O_3 and TiO_2 in grams

7. CaO

$$\frac{\text{Weight of precipitate, grams}}{\text{Oven-dry weight, grams}} \times 100 = \% \text{CaO}$$

8. MgO

$$\frac{\text{Weight of precipitate, grams}}{\text{Oven-dry weight, grams}} \times 36.2 = \% \text{MgO}$$

DISCUSSION

A semi-quantitative spectrographic analysis is run on each soil sample prior to the wet chemical analysis. This determines the approximate concentrations of the desired constituents in the soil and serves three useful purposes.

First, it enables one to choose the appropriate sample weight for the wet chemical analysis. Second, it reveals trace metals present in the soil and their concentrations. Third, it makes it possible to find other minor constituents that might otherwise be considered trace elements in the soil.

Appendix D

DTA THERMAL PATTERNS OF SOIL SAMPLES

A DTA thermal pattern (curve) is a thermal fingerprint of a substance as it is heated (or cooled) from one temperature level to another. Specifically, DTA is a technique of recording the difference in temperature between a substance (sample for analysis) and a reference material (thermally inert) as the two specimens are subjected to identical temperature regimes in an environment heated or cooled at a controlled rate. In general, deviations of the curve from a horizontal-like baseline indicate the following: (1) an upward (exothermic) movement or peak shows that energy (heat) is given off, and (2) a downward (endothermic) movement or peak shows that heat is absorbed or needed to accomplish a physical/chemical action or reaction.

The DTA curves were obtained on sample sizes of approximately 35 mg; the heating rate was a nominal 5°C per minute and the upper temperature limit was 1000°C.

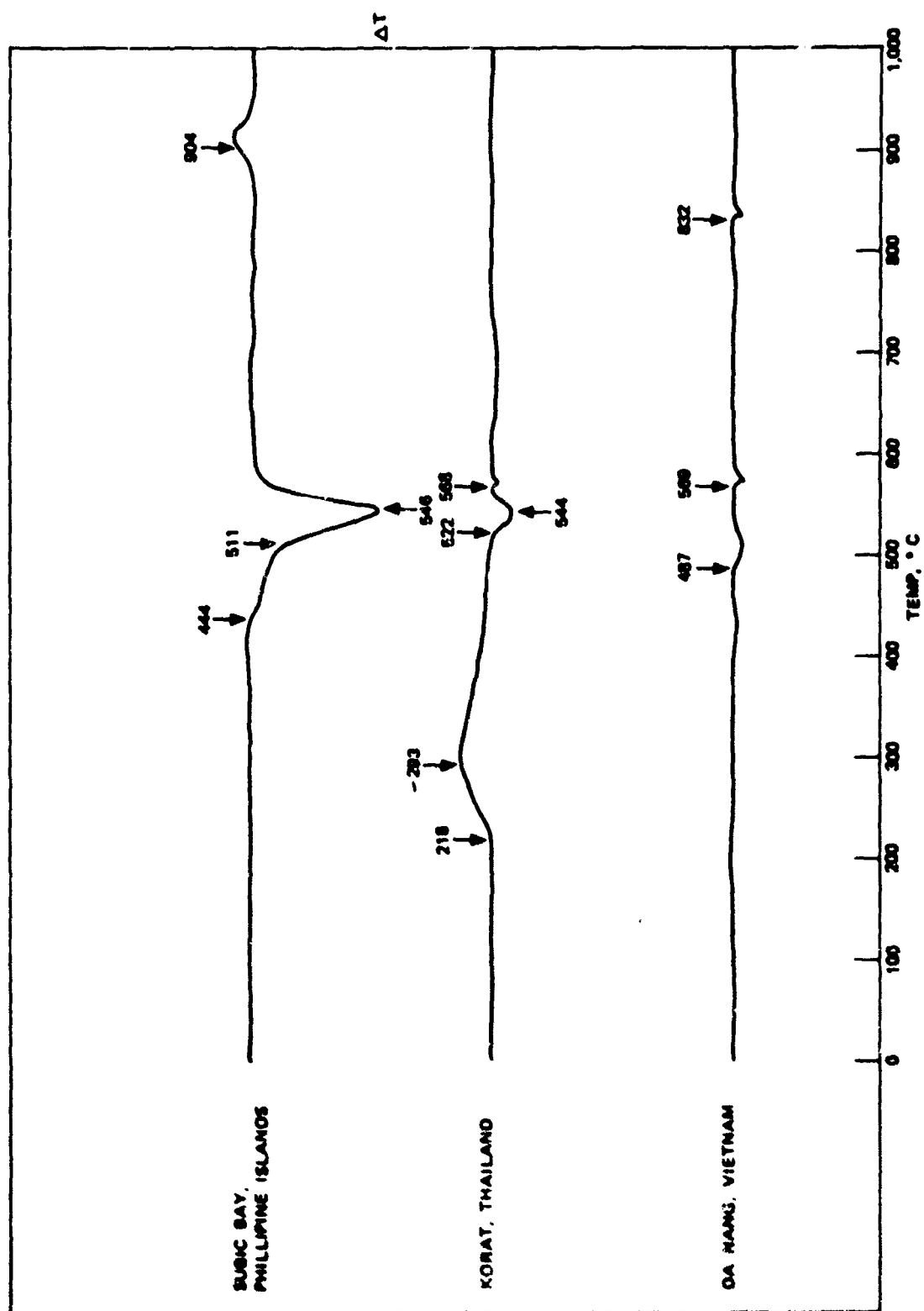
The DTA curves can be used with the other techniques in the identification of soil samples. For example, there are two endothermic phenomena which are readily recognizable in the thermal patterns. The first is the endotherm due to the dehydroxylation of the kaolinite, and the second is the endotherm due to the quartz transition.

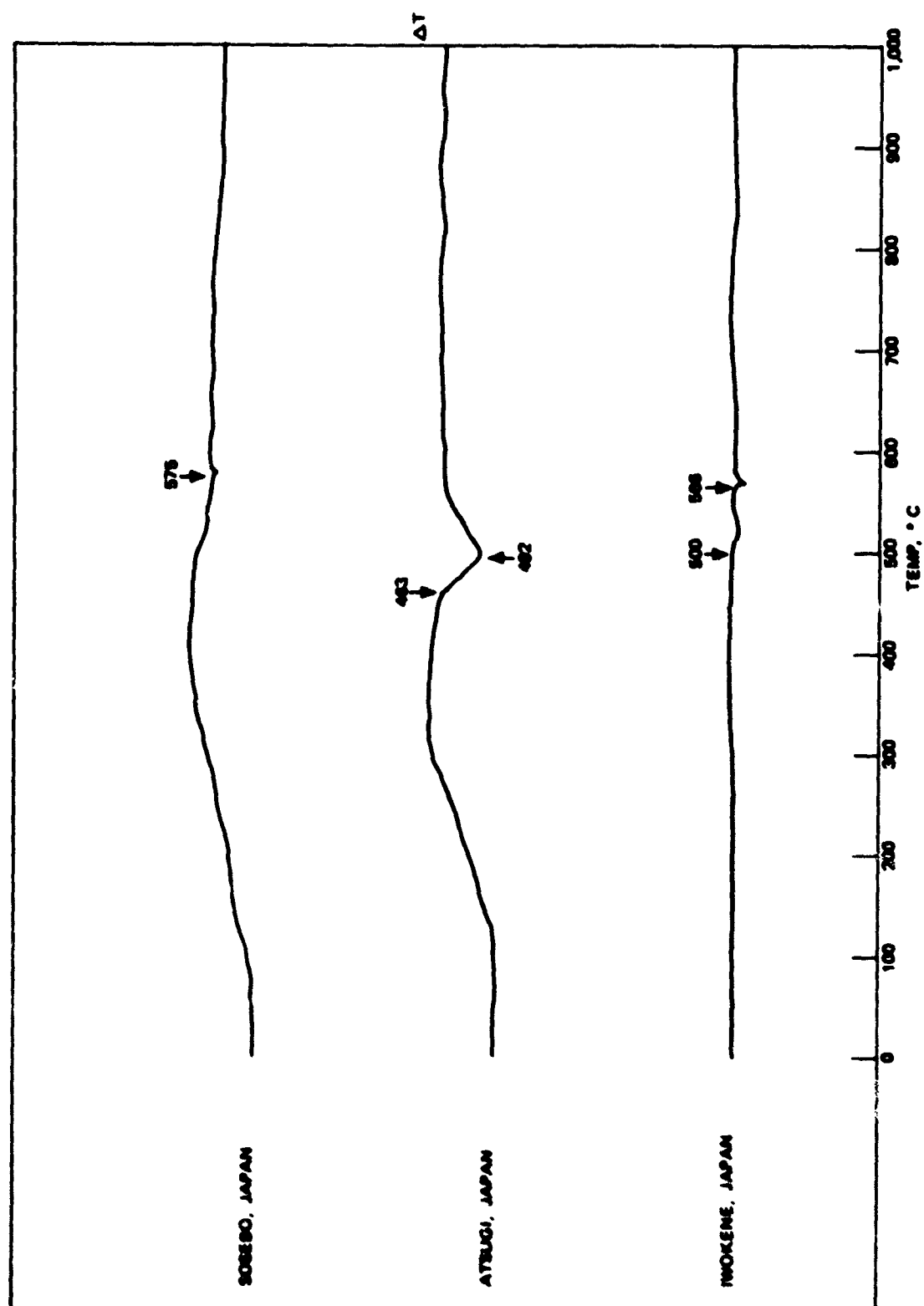
Kaolinite ($\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2 \cdot 2\text{H}_2\text{O}$) is the main constituent of China clay and of some other clays. When it undergoes dehydroxylation, the DTA curve shows a broad endothermic reaction in the temperature range of 500 to 550°C. This fact is illustrated; for example, in the thermal patterns on the samples from Subic Bay, Thailand, Coco Sola, and Ft. Clayton.

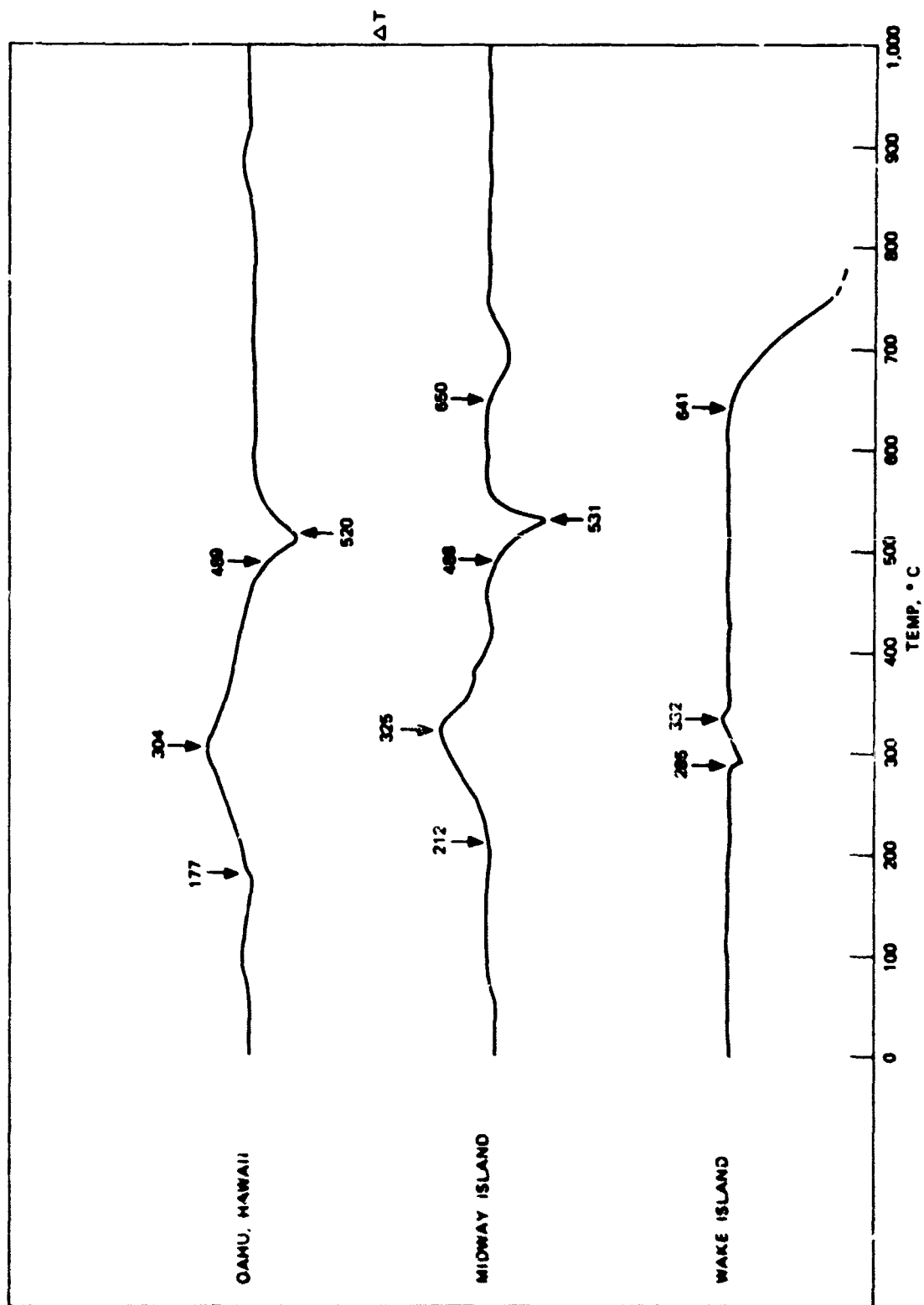
The presence of quartz (crystallized silica) can be detected in the soil sample by the appearance of a specific endotherm in the DTA curve. When quartz is heated within a definite temperature region, it undergoes an inversion or crystal rearrangement. In the majority of the DTA curves, the inversion temperature range was 569 to 576°C. This transition temperature can be seen, for example, in the thermal patterns of Da Nang, Eglin Air Force Base, Naval Weapons Center, and of Tanana Valley.

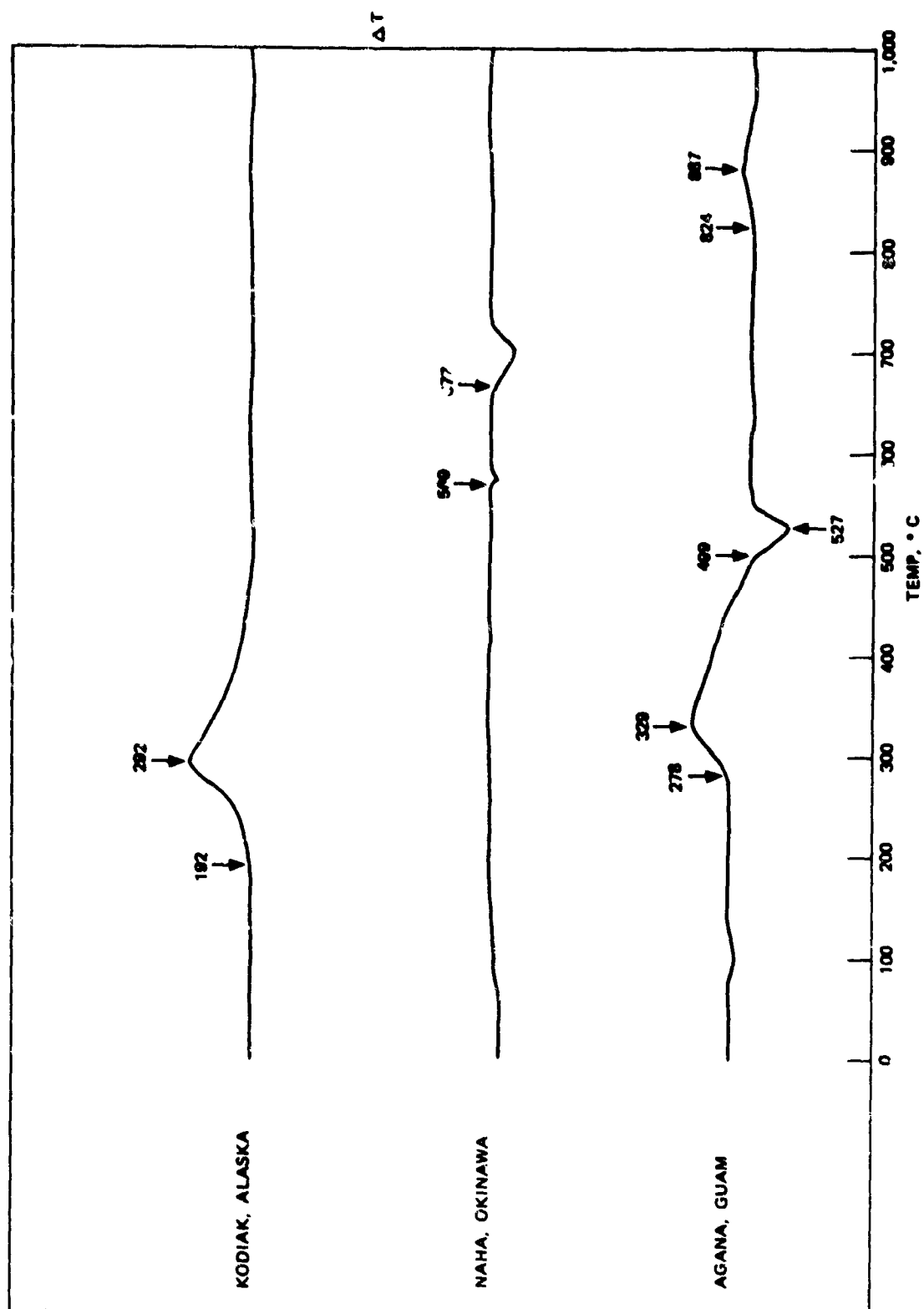
An area of activity is apparent in the approximate region of 200 to 450°C. These exothermic reactions are probably largely due to the decomposition of organic matter. This is illustrated, for example, in the thermal patterns of Thailand, Adak-2, Sea-Tac, and Guatemala City. The supposition is substantiated by the high ignition-loss values given in Table 1.

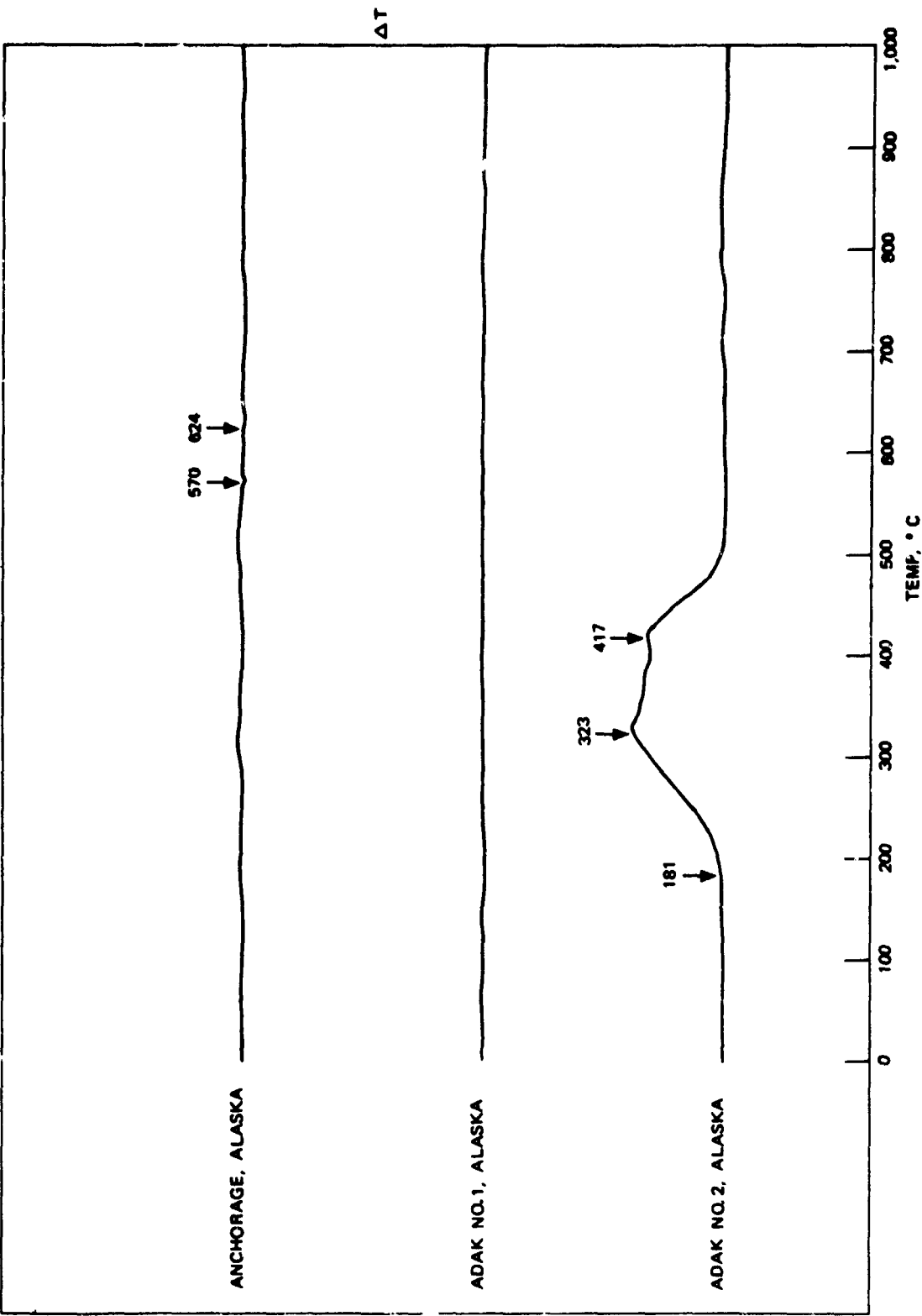
The decomposition of inorganic matter may be detected in the thermal patterns. For example, the extensive endothermic reaction in the 625 to 800°C region is due to the decomposition of calcite, the most common form of calcium carbonate. This is shown in the thermal patterns on the samples from Wake Island and Bermuda. The validity of the data is supported by the high ignition-loss values and the high percentage of calcium oxide (~50%) reported in Table 1. The calcium oxide is the final solid end-product from the decomposition of calcium carbonate.

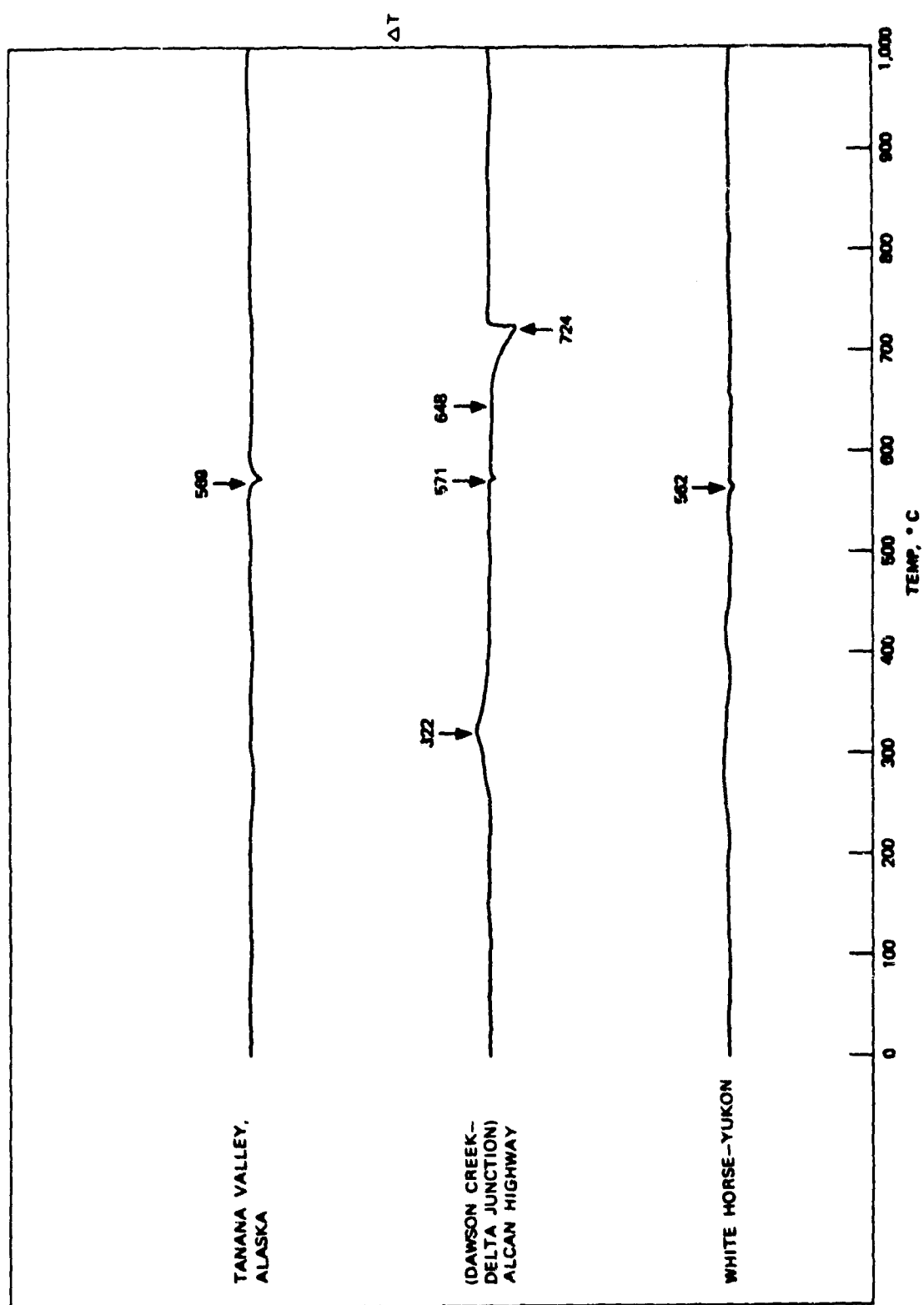


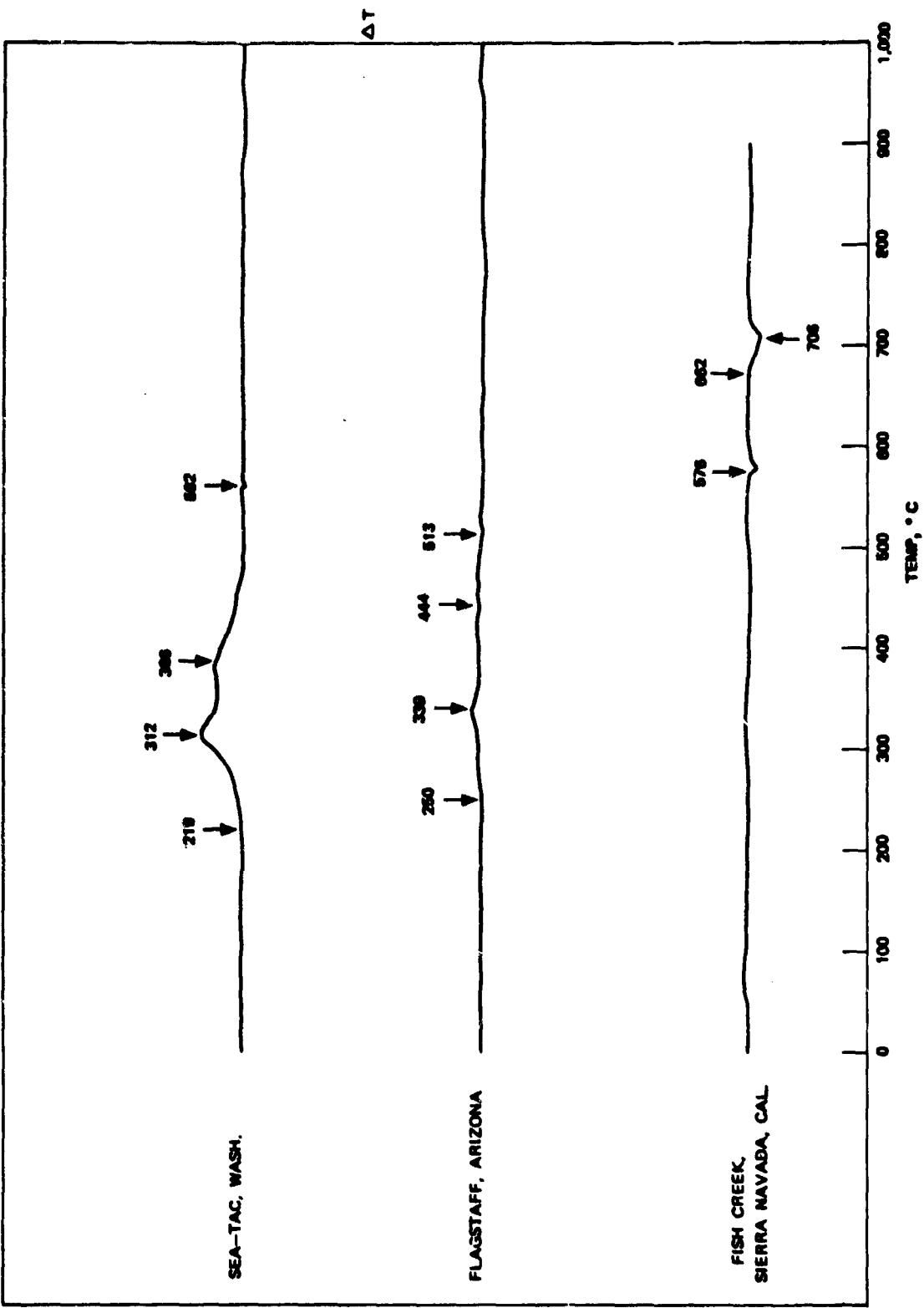


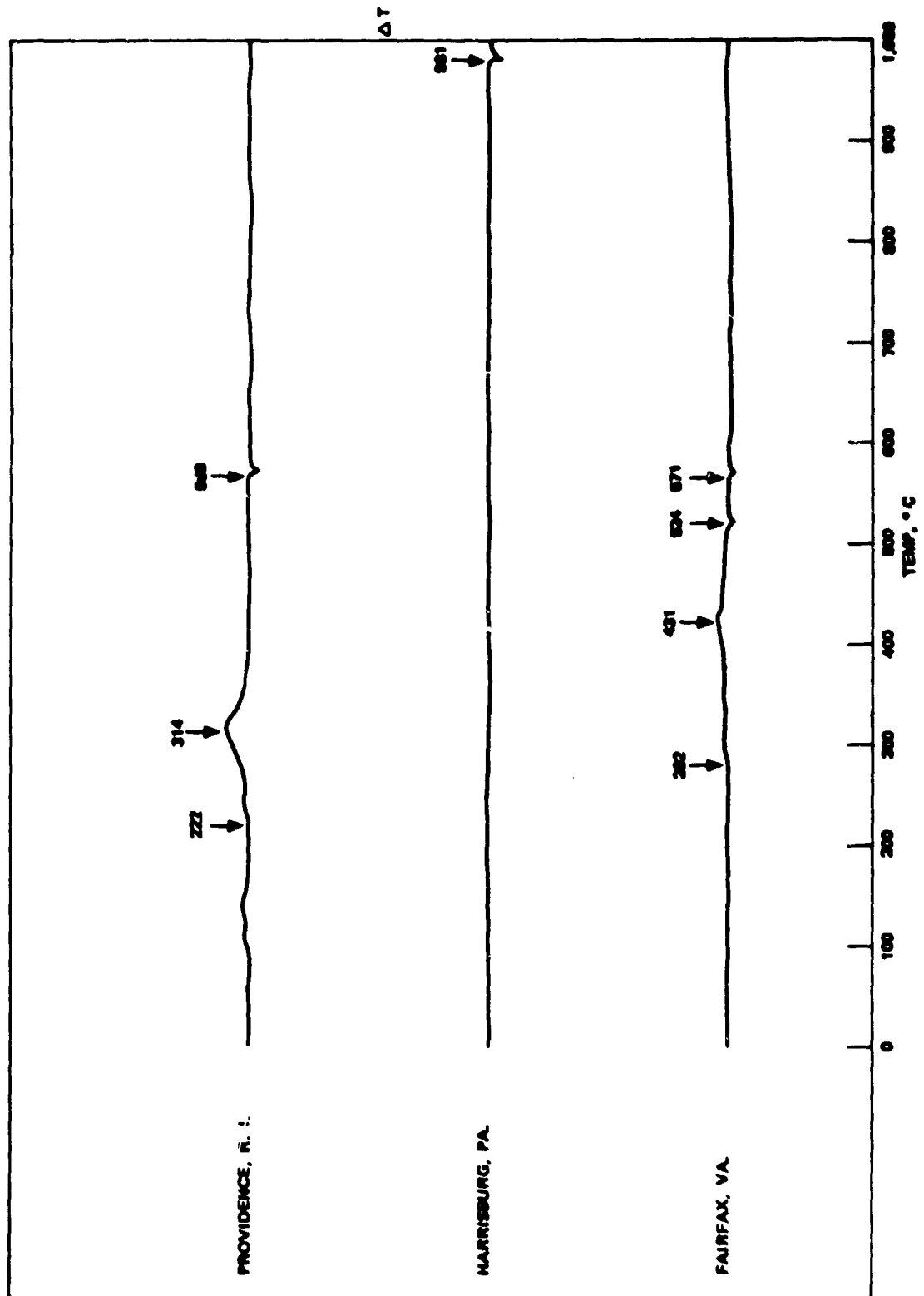


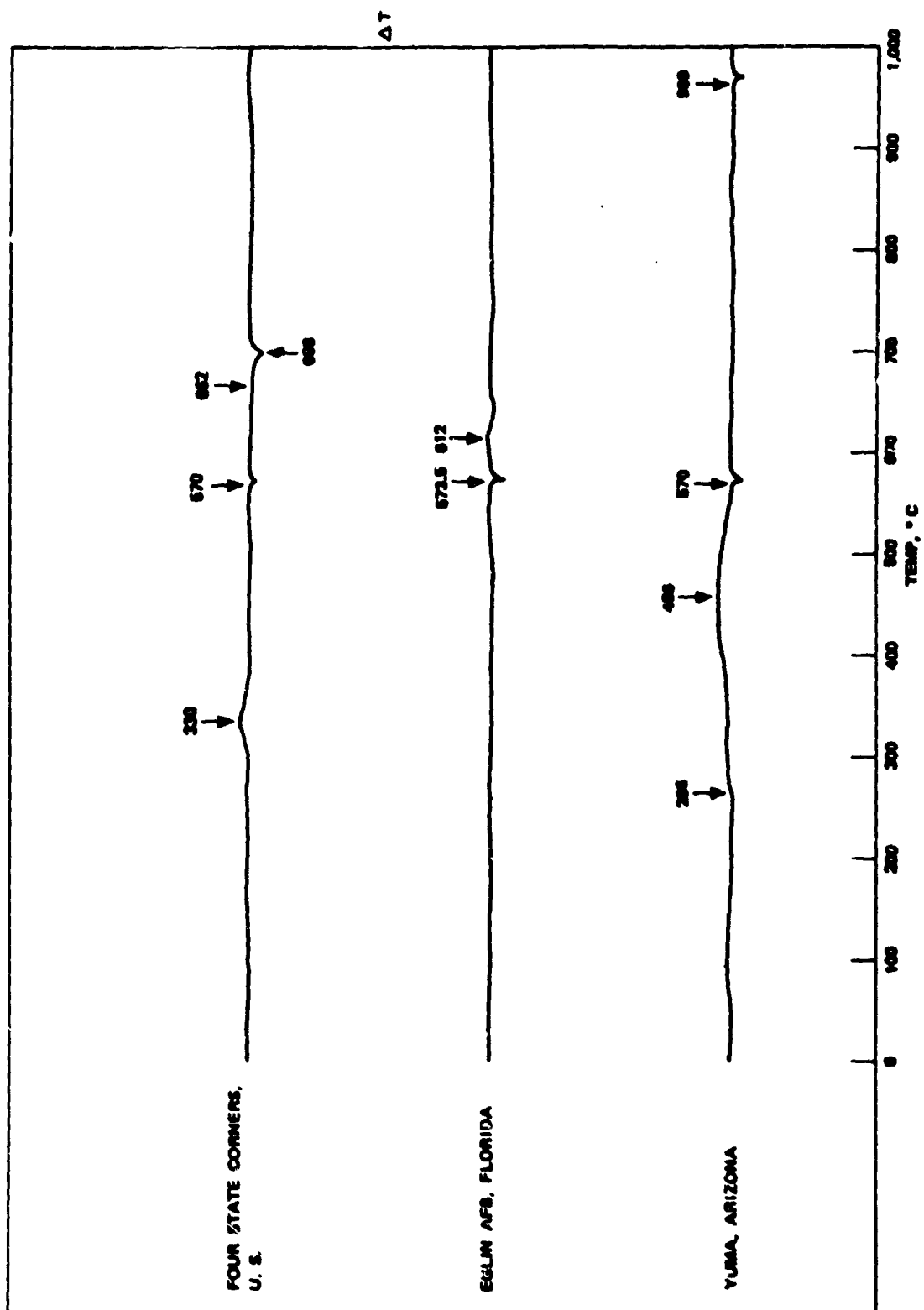


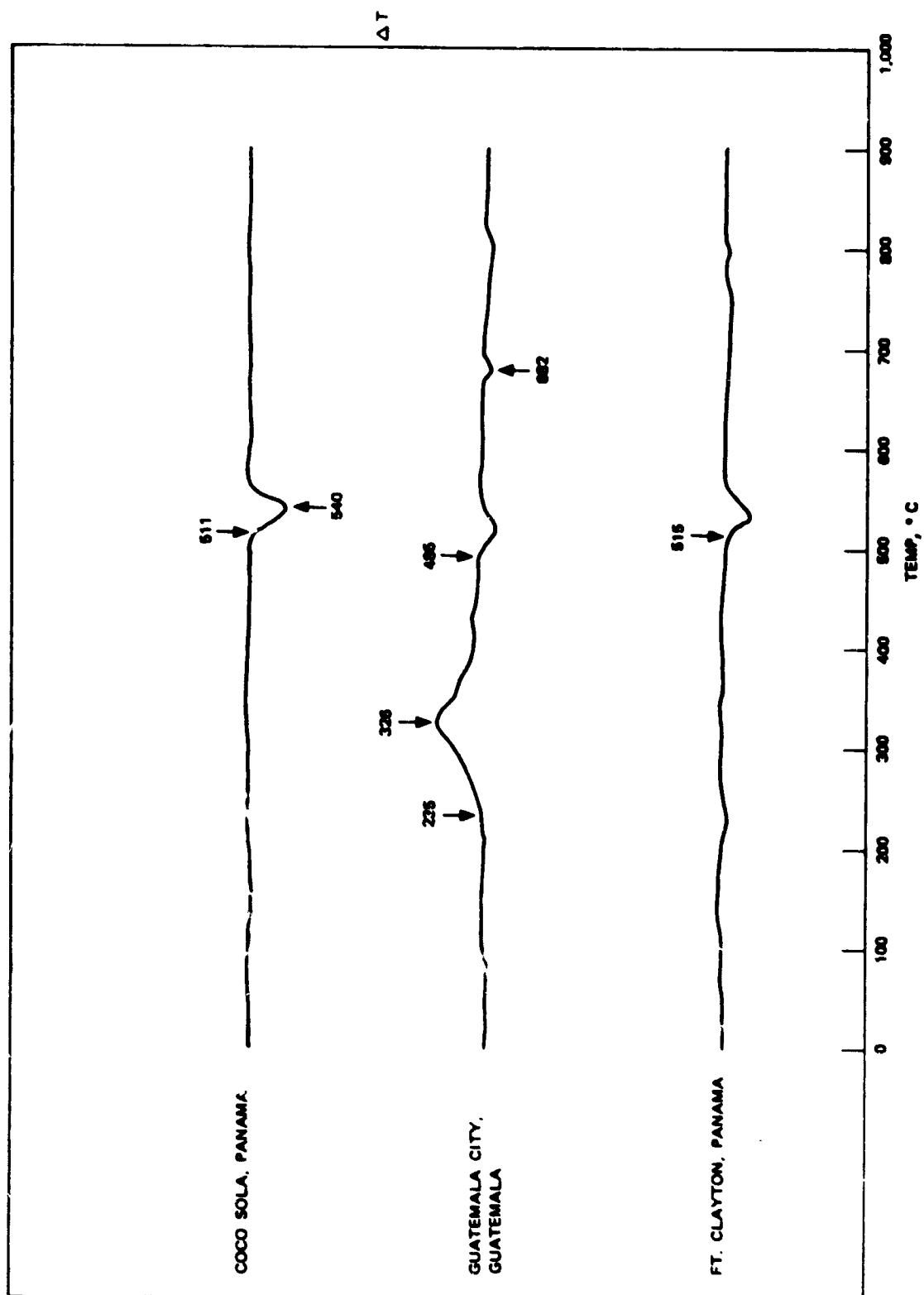


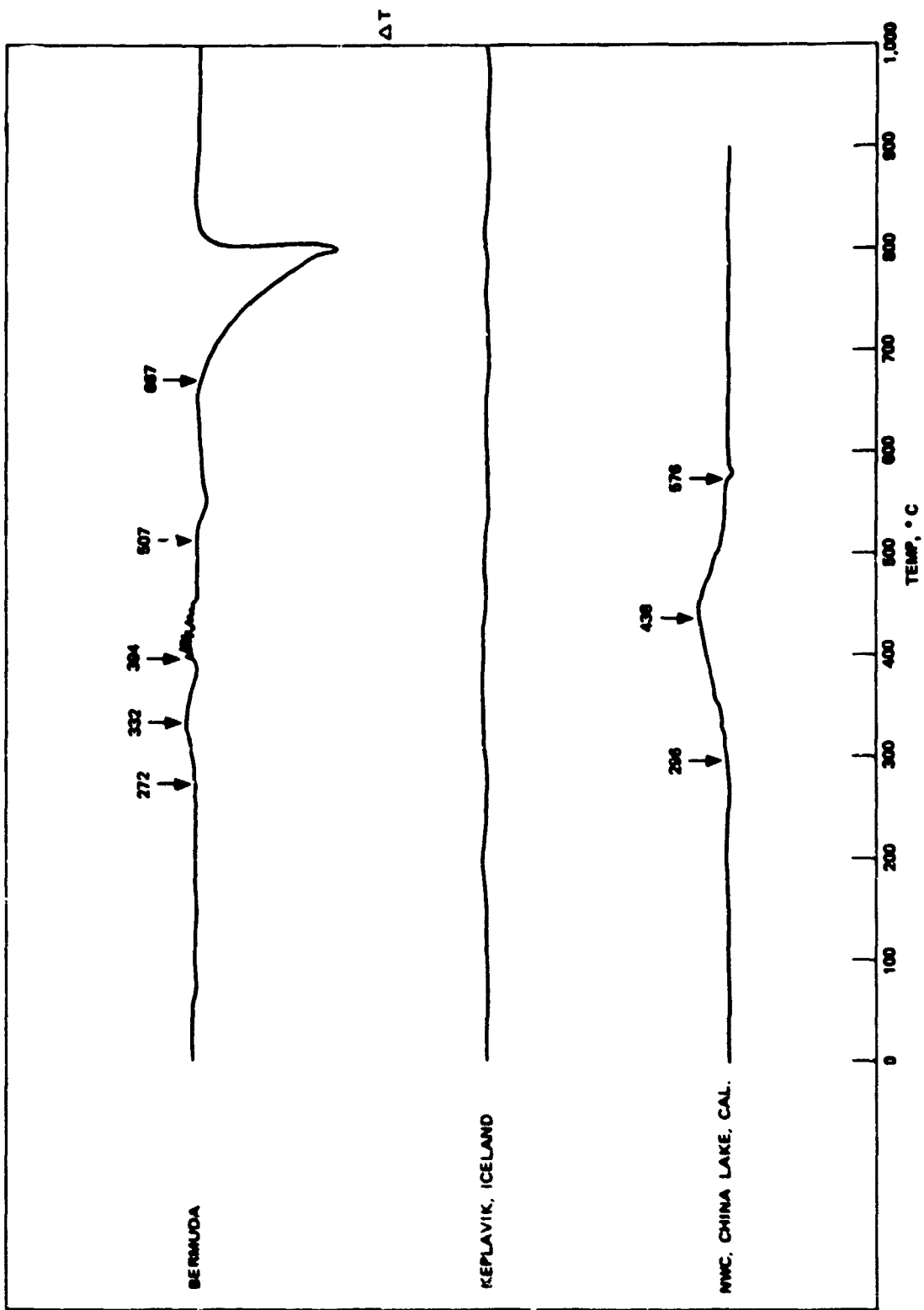












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